



**Final Quality Control Plan**  
**Remedial Investigation and Feasibility Study**  
**of the**  
**Defense Property Disposal Office**  
**Fort George G. Meade, Maryland**

Submitted to

U.S. Army Environmental  
Center (USAEC)  
Aberdeen, Maryland

May 1995

Prepared by:

Engineering Technologies Associates, Inc.  
3458 Ellicott Center Drive #101  
Ellicott City, MD 21043

DACA31-92-D-0045  
Delivery Order 0010

**Distribution Unlimited,  
approved for Public Release**

20061219194

## TABLE OF CONTENTS

<b>1.0</b>	<b>PROJECT DESCRIPTION</b>	<b>1</b>
1.1	Introduction	1
1.2	Site Background	1
1.2.1	Site Description	3
1.2.2	Site History	3
1.3	Task Objectives and Scope of Work	3
1.4	Application of the Project QC Plan	5
1.5	Organization of Document	5
<b>2.0</b>	<b>PROJECT AND QA/QC ORGANIZATION AND RESPONSIBILITIES</b>	<b>8</b>
2.1	Project Organization	8
2.1.1	Program Manager	8
2.1.2	Remedial Investigation Project Manager	8
2.1.3	Feasibility Study Project Manager	8
2.1.4	Additional Staff	8
2.2	Engineering Technologies Associates, Inc. QA/QC Organization	10
2.2.1	Program QA Officer	10
2.2.2	Lead Chemist	11
2.3	DataChem Project QA/QC Organization	11
<b>3.0</b>	<b>QA OBJECTIVES FOR MEASURING DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS AND COMPARABILITY</b>	<b>12</b>
3.1	Introduction	12
3.2	QA Objectives for the DRMO Project	12
3.2.1	Precision	19
3.2.2	Accuracy	19
3.2.3	Representativeness	19
3.2.4	Completeness	19
3.2.5	Comparability	20
<b>4.0</b>	<b>SAMPLE COLLECTION</b>	<b>21</b>
4.1	Sampling for the Remedial Investigation at the DRMO Yard	21
4.1.1	Subsurface Soil Sampling	22
4.1.2	Ground Water Sampling	22
4.1.3	Surface Runoff/Sediment Sampling	22
4.1.4	Sediment Sampling	23
4.2	Location and Elevation Survey	23
4.3	Investigation-Derived Wastes	23
4.4	Sample Containers, Preservation and Handling	24
4.4.1	Sample Containers	24

	4.4.2 Sample Preservation and Holding Times . . . . .	24
4.5	Field Quality Control Samples . . . . .	24
4.6	Sample Handling . . . . .	24
5.0	SAMPLE CUSTODY . . . . .	30
5.1	Field Custody Procedures . . . . .	30
5.2	Laboratory Custody Procedures . . . . .	31
6.0	CALIBRATION PROCEDURES AND FREQUENCY . . . . .	32
6.1	Field Instrumentation . . . . .	32
6.2	Laboratory Calibration . . . . .	32
7.0	ANALYTICAL PROCEDURES . . . . .	33
7.1	Analytical Program . . . . .	33
7.3	Analyst Qualification . . . . .	34
7.4	Field Analytical Methods . . . . .	34
8.0	DATA REDUCTION, VALIDATION, AND REPORTING . . . . .	35
8.1	Engineering Technologies Associates, Inc. Data Management . . . . .	35
	8.1.1 Flow of Map Data into IRDMIS . . . . .	35
	8.1.2 Flow of Analytical Data into IRDMIS . . . . .	35
8.2	Data Reduction . . . . .	35
8.3	Data Validation . . . . .	36
8.4	Data Validation Procedures . . . . .	37
8.5	IRDMIS Record and Group Checks . . . . .	38
8.6	Data Reporting . . . . .	39
9.0	INTERNAL QC CHECKS AND FREQUENCY . . . . .	40
9.1	Control Samples . . . . .	40
9.2	Field Control Samples . . . . .	40
	9.2.1 Trip Blanks . . . . .	40
	9.2.2 Field Equipment/Rinsate Blanks . . . . .	40
9.3	Laboratory Control Samples . . . . .	41
	9.3.1 Laboratory Blanks . . . . .	41
	9.3.2 Laboratory Duplicates . . . . .	42
	9.3.3 Calibration Standards . . . . .	42
	9.3.4 Spike Sample . . . . .	42
	9.3.5 Internal Standard . . . . .	42
9.4	Concentration and Frequency of Control Samples . . . . .	42
	9.4.1 Class 1 Performance Demonstrated Method . . . . .	43
	9.4.2 Class 1A Performance Demonstrated Method (GC/MS only) . . . . .	44
9.5	Data Reporting for Quality Control . . . . .	44
	9.5.1 Class 1, Class 1A, and Class 1B Performance Demonstrated Methods . . . . .	44

<b>10.0</b>	<b>PERFORMANCE AND SYSTEM AUDITS</b>	<b>46</b>
10.1	Field Audits	46
10.2	Laboratory Audits	47
10.2.1	Data Review	48
10.3	Project Audits	48
<b>11.0</b>	<b>PREVENTIVE MAINTENANCE</b>	<b>49</b>
11.1	Field Instruments	49
11.2	Laboratory Equipment	49
<b>12.0</b>	<b>PROCEDURES TO ASSESS DATA ACCURACY, PRECISION, AND COMPLETENESS</b>	<b>50</b>
12.1	Lack of Fit (LOF) and Zero Intercept (ZI) Tests	50
12.2	Certified (Performance Demonstrated) Reporting Limit (CRL)	50
12.3	Method Performance Demonstration Accuracy	52
12.4	Method Performance Demonstration Standard Deviation	53
12.5	Method Performance Demonstration Percent Inaccuracy	53
12.6	Method Performance Demonstration Percent Imprecision	53
12.7	Data Moving-Average Accuracy and Precision	54
12.8	Control Charts	56
12.8.1	Control Chart Plotting: Single-Day	56
12.8.2	Three-Point Moving Average	57
12.9	Out-of-Control Conditions	58
12.10	Non-AEC Methods	58
12.11	Completeness	59
<b>13.0</b>	<b>CORRECTIVE ACTIONS</b>	<b>60</b>
13.1	Field Situations	61
13.2	Laboratory Situations	61
<b>14.0</b>	<b>QUALITY ASSURANCE REPORTS TO MANAGEMENT</b>	<b>62</b>
14.1	Laboratory Reports	62
14.2	Program QA Officer and Lead Chemist Reports	63

## List of Acronyms and Abbreviations

ADD	Average Daily Dose
AEHA	Army Environmental Hygiene Agency
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing and Materials
ATSDR	Agency for Toxic Substances Disease Registry
BRAC	Base Closure and Realignment Act
CAR	Corrective Action Report
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COE	Corps of Engineers
COR	Contracting Officer's Representative
CRAVE	Carcinogen Risk Assessment Verification Endeavor
CRL	Certified Reporting Limit
DCE	1,1-Dichloroethene
DPDO	Defense Property and Disposal Office
DRMO	Defense Reutilization and Marketing Office
EIS	Environmental Impact Study
EPA	United States Environmental Protection Agency
ETA	Engineering Technologies Associates, Inc.
FGGM	Fort George G. Meade
FS	Feasibility Study
FSPM	Feasibility Study Project Manager
GC/MS	Gas Chromatography/Mass Spectrometry
GC	Gas Chromatography
GFAA CV	Graphite Furnace and Cold Vapor Atomic Absorption
GPM	Gallons Per Minute
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HEAST	Health Effects Assessment Summary Tables
HI	Hazard Index
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Argon Plasma Emission Spectroscopy
IR	Installation Restoration
IRDMIS	Installation Restoration Data Management Information System
IRIS	Integrated Risk Information Systems
IRM	Interim Reference Materials
L	Liter
LCL	Lower Control Limit

LOF	Lack of Fit
LWL	Lower Warning Limit
MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goal
MDE	Maryland Department of the Environment
MS	Mass Spectrometry
NAD27	North American Datum 1927
NCP	National Contingency Plan
NEPA	National Environmental Policy Administration
NIST	National Institute of Standards and Technology
No.	Number
NPDWR	National Primary Drinking Water Regulations
NPL	National Priorities List
ODC	Other Direct Costs
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PE	Professional Engineer
PCB	Polychlorinated Biphenyl
PCE	Perchloroethene
PID	Photoionization Detector
pH	Percent Hydroxide
PP	Proposed Plan
PQL	Practical Quantitative Limit
PRI	Potomac Research, Inc.
PVC	Polyvinyl Chloride
QA/QC	Quality Assurance/Quality Control
QAC	Quality Assurance Coordinator
QAP	Quality Assurance Plan
QCP	Quality Control Plan
PRI	Potomac Research, Inc.
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
RI	Remedial Investigation
RIA	Remedial Investigation Addendum
RIPM	Remedial Investigation Project Manager
RPD	Relative Percent Difference
ROD	Record of Decision
SARM	Standard Analytical Reference Material
SI	Site Inspection
SIA	Site Investigation Addendum
SOPs	Standard Operating Procedures
SLI	Site Location Identity
SQL	Sample Quantitation Limit
SVOC	Semivolatile Organic Compound

TAL	Target Analyte List
TCA	Tetrachloroethane
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TEPS	Total Environmental Program Support
TPHC	Total Petroleum Hydrocarbon
TQM	Total Quality Management
TWP	Technical Work Plan
UCL	Upper Control Limit
USAEC	United States Army Environmental Center
USATHAMA	United States Army Toxic and Hazardous Materials Agency
USC	Unique Sample Code
UWL	Upper Warning Limit
UXO	Unexploded Ordnance
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WCFS	Woodward-Clyde Federal Services
ZI	Zero Intercept

## **1.0 PROJECT DESCRIPTION**

### **1.1 Introduction**

This Quality Control Plan (QCP) was prepared by Engineering Technologies Associates, Inc. (ETA) under Contract No. DACA31-92-D-0045, Delivery Order 0010, for the U. S. Army Environmental Center (USAEC) to address the Remedial Investigation and Feasibility Study of the Defense Reutilization and Marketing Office (DRMO) Yard at Fort Meade, formerly referred to as the Defense Property and Disposal Office (DPDO). This Quality Control Plan (QCP) has been developed in accordance with the United States Toxic and Hazardous Materials Agency (USATHAMA) Geotechnical Requirements, and the USATHAMA Quality Assurance Program and the Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.

This QCP for the RI/FS at the DRMO Yard has been developed to comply with the requirements of the USAEC Quality Assurance Program, USATHAMA PAM 11-41, Revision No. 0, January 1990 and appropriate EPA Region III Quality Assurance Guidance as applicable. Our subcontracted laboratory will be DataChem Laboratories, of Salt Lake City, Utah. They will provide chemical analyses of environmental samples collected during this investigation. Therefore the QA Program Plan from DataChem Laboratories is included as Attachment A.

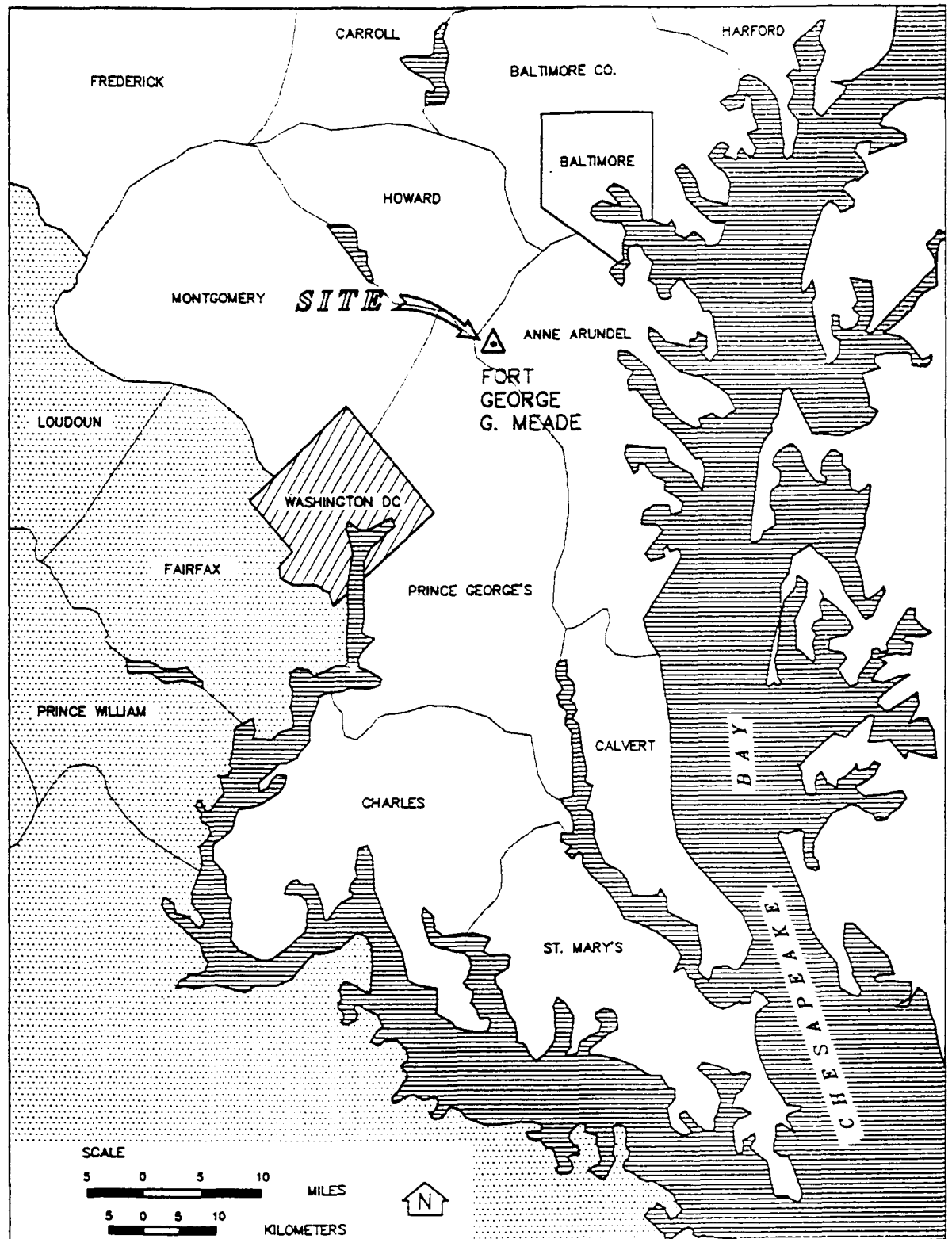
ETAs corporate policy includes a commitment to a high standard of quality in the work it performs for and delivers to its clients. This policy is reflected in the quality of our general operating policies and procedures and the quality of the workmanship that is produced for our clients. We expect the same level of commitment to quality from our subcontractors.

The objective of the USAEC Quality Assurance Program is to establish a QA system and proper QC procedures associated with the Quality Control Plan for specific projects, such as the Remedial Investigation and Feasibility Study at the DRMO Yard at Fort Meade. USAEC defines QA as "the system whereby an organization provides assurance that monitoring of quality related activities has occurred"; QC as "specific actions taken to ensure that system performance is consistent with established limits". It is these actions which ensure accuracy, precision and comparability of results. This project specific QC Plan is developed to establish the procedures that must be adhered to in order to ensure adequate quality to support decisions regarding potential remedial actions.

### **1.2 Site Background**

The DRMO Yard is located off Remount Road south of Rock Avenue and it abuts State Route 32 to the south (Figure 1). The site covers approximately 8.7 acres and is used as a storage area for various equipment, including vehicles, transformers, electronic equipment, heating and cooling units, pipes, dumpsters, and scrap metals (A.D. Little, 1994).





SOURCE: ESE, 1986

DESIGNED	EM.	10/84
DRAWN	S.P.	10/84
CHECKED	LL	10/84
APPROVED	DK	10/84

# ENGINEERING TECHNOLOGIES ASSOCIATES, INC.

ENGINEERS • PLANNERS • SURVEYORS

3408 ELLICOTT CENTER DRIVE SUITE 101  
ELLICOTT CITY, MARYLAND 21043  
BAL. (410) 528-1100 FAX (410) 528-1101

Figure 1  
FORT GEORGE G. MEADE  
LOCATION MAP

SCALE: AS SHOWN CONTRACT NO. 92307.010 DATE: OCT. 13, 1994 SHEET

Previous studies (EA Engineering, and Science, Inc. 1992 and A. D. Little, Inc. 1993) have detected contamination in ground water in monitoring wells located along the northern side of Route 32 (southern boundary of the DRMO Yard). The objective of this study is to determine if that contamination has migrated beneath Route 32 onto the lands to the south. These lands were transferred to the Department of Interior/Pautextent Environmental Science Center in 1991 and 1992.

#### 1.2.1 Site Description

Fort Meade is located in Anne Arundel County, MD, between Washington, D.C. and Baltimore. The entire installation includes approximately 5,000 acres and the closest city is Odenton, MD (Figure 1). Fort Meade has been in operation since 1917 and the current workforce includes approximately 20,000 people (A. D. Little, 1994).

The DRMO Yard is located along Remont Road south of Rock Avenue immediately north of State Route 32 (Figure 2). The site covers an area of approximately 8.7 acres and is a storage area for various equipment, including vehicles, transformers, electronic equipment, heating and cooling units, pipes, dumpsters, and scrap metals (A. D. Little, 1994).

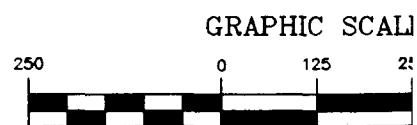
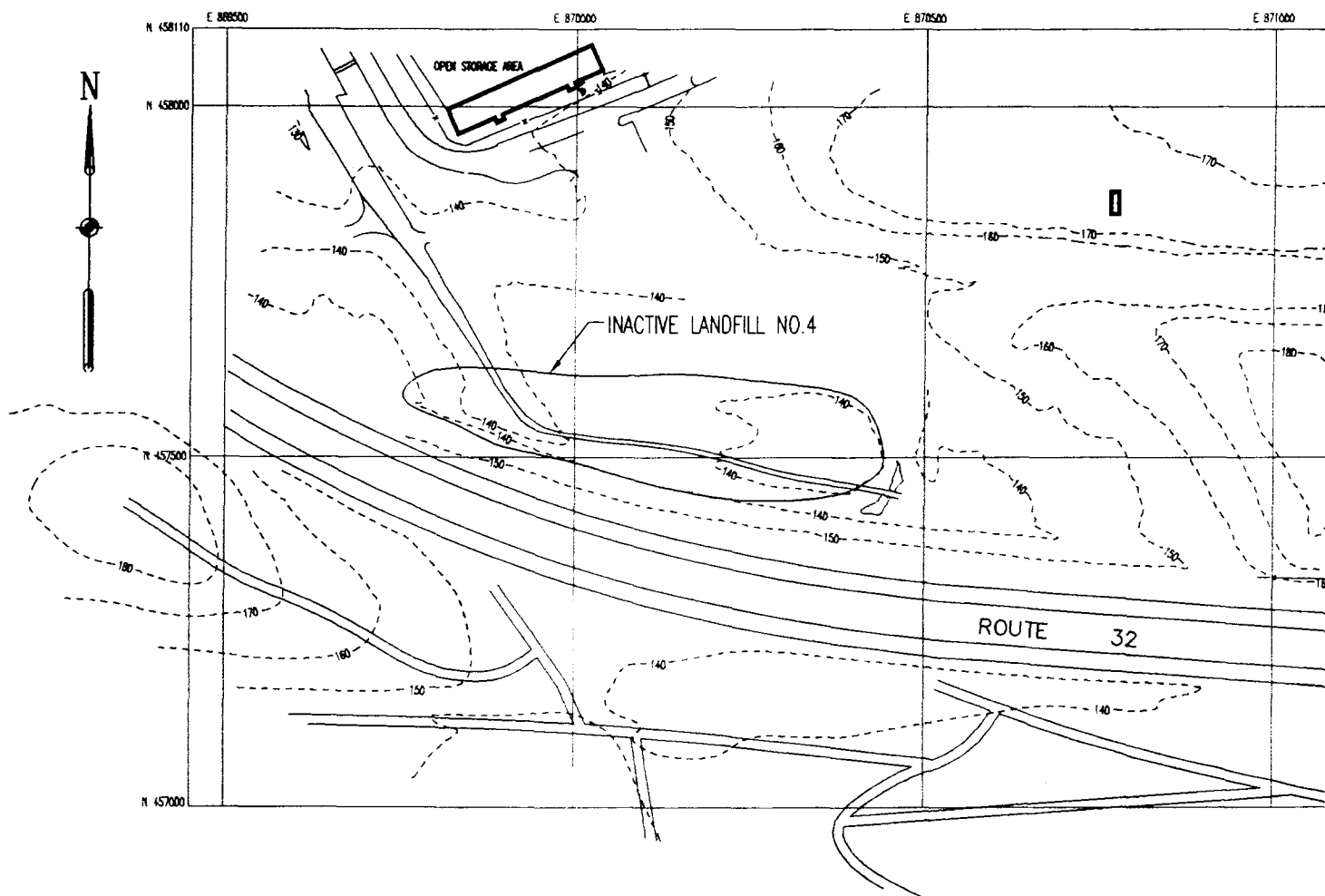
#### 1.2.2 Site History

The two previous studies were undertaken in an attempt to determine the extent of contamination at the DRMO Yard and whether that contamination may be migrating. These studies have concluded that ground water beneath the site is flowing toward the north/northeast and that the ground water is contaminated with volatile organics and metals, some above benchmarks. However, there is now some indication that the ground water may be flowing to the south beneath State Route 32 onto a land parcel which was transferred to another Federal agency. This indication is due to the anomolous water level readings in one on-site monitoring well along the southern boundary of the site. Also the highest levels of contaminant concentrations have been detected along this southern boundary.

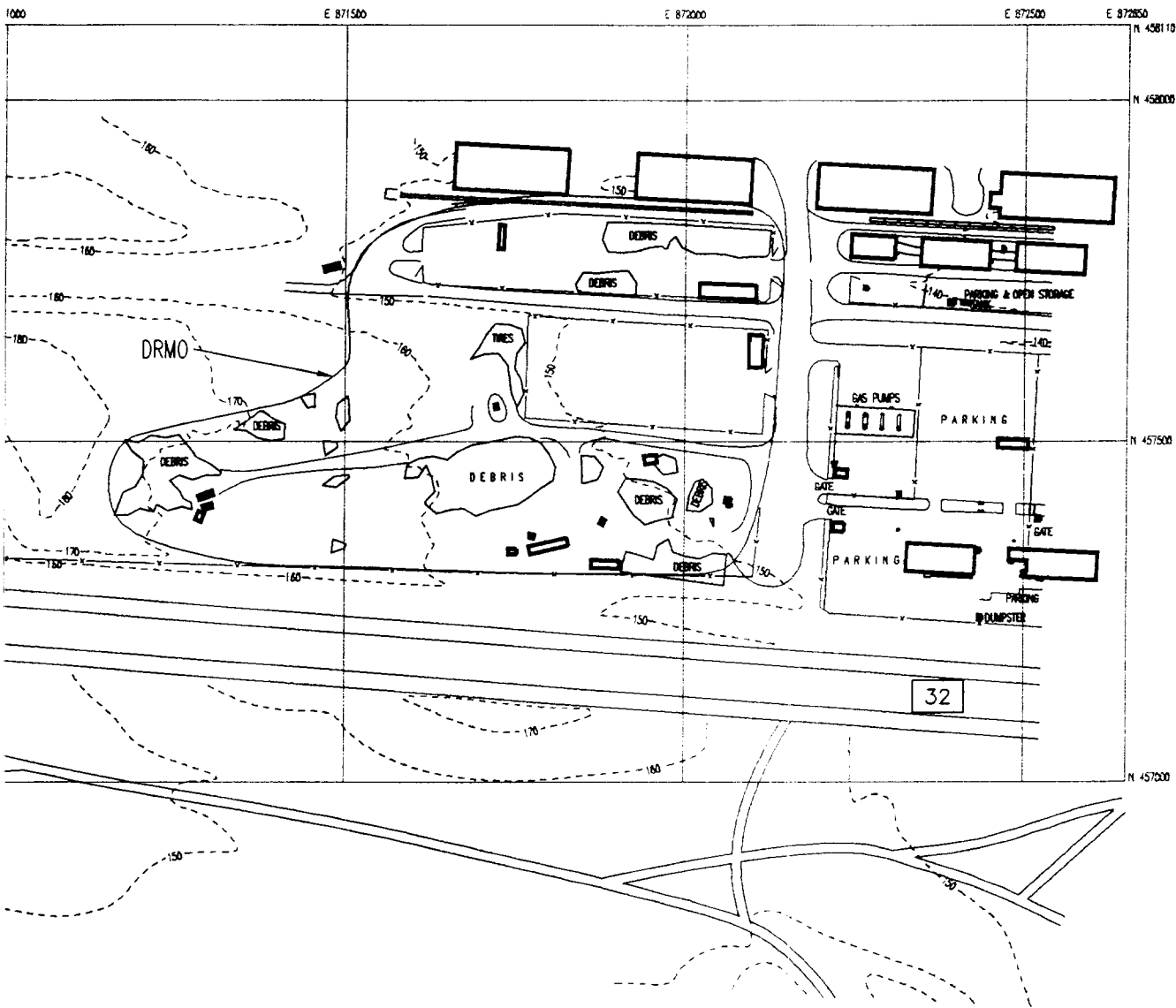
### 1.3 Task Objectives and Scope of Work

The objective for this task is to determine whether ground water or surface runoff is migrating from the DRMO site to the BRAC land parcel south of State Route 32. This will be accomplished by performing a Remedial Investigation. If it is determined that this potential migration is occurring, then a Feasibility Study will be undertaken to determine the most appropriate method to remediate the contamination migration.

The scope of work for this investigation is based on USAEC's Request for Proposal for a delivery order for Fort George G. Meade Remedial Investigation/Feasibility Study - Defense Property Disposal Organization. The scope includes the following tasks:

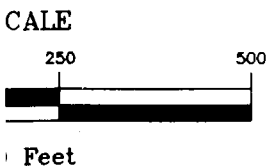


1 Inch = 250 Feet



# LEGEND

--- 160 --- TOPOGRAPHIC CONTOUR



2

FIGURE NO. 2

## **DRMO** SITE LOCATION FORT MEADE, MARYLAND

ENGINEERING TECHNOLOGIES  
ASSOCIATES, INC.

ENGINEERS \* PLANNERS \* SURVEYORS

3458 ELLICOTT CENTER DRIVE, SUITE 101  
ELLICOTT CITY, MARYLAND 21043  
BALTO. 481-8920 WASH. 621-4890

DESIGNED	K.W.	10/94
DRAWN	S.P.	0/94
CHECKED	K.W.	10/94
APPROVED	D.K.	10/94

SCALE: AS SHOWN CONTRACT NO: 92307.010 DATE: 10/94 SHEET: 1

- Site Reconnaissance and Data Review
- Project Plans
- Project Meetings
- Remedial Investigation
- Feasibility Study
- Final Reporting
- Management and Cost Reporting

RI activities include the following:

- UXO screening of well locations and borings
- Installation of two ground water monitoring wells south of State Road 32
- Soil sampling during well completion
- Surveying of wells
- Aquifer testing to determine ground water flow direction
- Ground water sampling and analyses
- Surface runoff and sediment sampling
- Limited risk assessment (human health and ecological)

#### **1.4 Application of the Project QC Plan**

This QCP has been written for both the analytical and field portion of the RI.

QA is a system that an organization implements to assure that monitoring of quality-related activities occurs. This is generally accomplished by implementation of a recordkeeping system for documentation of activities including traceability, completeness and security of documents. Implementation of the QA program in the field, at the office and at the laboratory ensures that decisions based on data or documents can be sustained. QC refers to the specific actions taken to verify that the organization's QA Program is being implemented. Through the QC actions accuracy, precision and comparability of results are achieved.

This QCP establishes the procedures to be followed during the performance of this task to ensure that USAEC QA goals are attained. This plan will establish procedures for use in field activities and generation of laboratory data. Specific instructions for environmental sampling, chemical analyses, chain-of-custody procedures, computer and document-related activities and final calculations will be described. DataChem Laboratories, ETA's subcontracted laboratory, will follow the procedures outlined in this Plan.

#### **1.5 Organization of Document**

This QCP has been prepared using the guidance provided in the USAEC QA Program Manual (January 1990); the sections of the Plan have been organized as per the guidance document.

**Section 1.0 Project Description:** discusses the site background and describes the site including the site history. It discusses the past investigative efforts and the current project scope.

**Section 2.0 Project and QA/QC Organization and Responsibilities:** discusses the organization of the project and identifies the responsibilities of each staff member in the organization. It also describes the role DataChem will play in the project.

**Section 3.0 QA Objectives for Measuring Data in Terms of Precision, Accuracy, Representativeness, Completeness and Comparability:** discusses the QA data objectives for all data collected as a result of this project.

**Section 4.0 Sample Collection:** describes the specific sampling procedures to be used during the collection of environmental samples.

**Section 5.0 Sample Custody:** describes the specific sample custody procedures to be implemented including field and laboratory custody procedures.

**Section 6.0 Calibration Procedures and Frequency:** describes the specific field and laboratory instrument calibration guidelines to be followed.

**Section 7.0 Analytical Procedures:** describes the procedures for field and laboratory data collection; most analytical procedures used during this project are USAEC methods.

**Section 8.0 Data Reduction, Validation and Reporting:** describes the procedures to be followed during data reduction, validation and reporting. These procedures conform to the USAEC IRDMIS requirements and USEPA Region III data validation guidance.

**Section 9.0 Internal QC Checks and Frequency:** describes the internal sampling and analysis activities and specifies the frequency of each.

**Section 10.0 Performance and System Audits:** describes the audits that need to be conducted during the progress of this project.

**Section 11.0 Preventive Maintenance:** describes the maintenance plan that DataChem Laboratories will implement to ensure instrumentation accuracy.

**Section 12.0 Procedures Used to Assess Data Accuracy, Precision and Completeness:** describes the specific procedures regularly used to ensure the accuracy, precision and completeness of data quality.

**Section 13.0 Corrective Actions:** describes the recommended corrective actions to be taken in both field and laboratory environments.

**Section 14.0 Quality Assurance Reports to Management:** describes the nature of QA reports to management.

## **2.0 PROJECT AND QA/QC ORGANIZATION AND RESPONSIBILITIES**

The organizational structure for the DRMO project will be discussed in this section. The structure of the organization indicates the overall assignment of project responsibility for all aspects of the project and the functional communication between the elements. The organizational diagram is presented in Figure 3. The actual roles of the key project personnel are described below:

### **2.1 Project Organization**

#### **2.1.1 Program Manager**

The Program Manager for this contract and project is Donald H. Koch, P.E. He will be responsible for monitoring technical progress, reviewing and approving all work products, reviewing and approving all project deliverables prior to their submittal to USAEC. He will also monitor the financial and schedule control and implement corrective action, if necessary.

#### **2.1.2 Remedial Investigation Project Manager**

The Remedial Investigation Project Manager (RIPM) for this project is Larry Lumeh, Ph.D. He will be responsible for project staffing and direct management of all staff assigned to this project. He will institute financial and schedule control and will review and approve all deliverables prior to their submittal to USAEC. He will maintain a liaison with the USAEC Project Officer and the Fort Meade Environmental Office, keeping them informed of the technical progress of the project.

#### **2.1.3 Feasibility Study Project Manager**

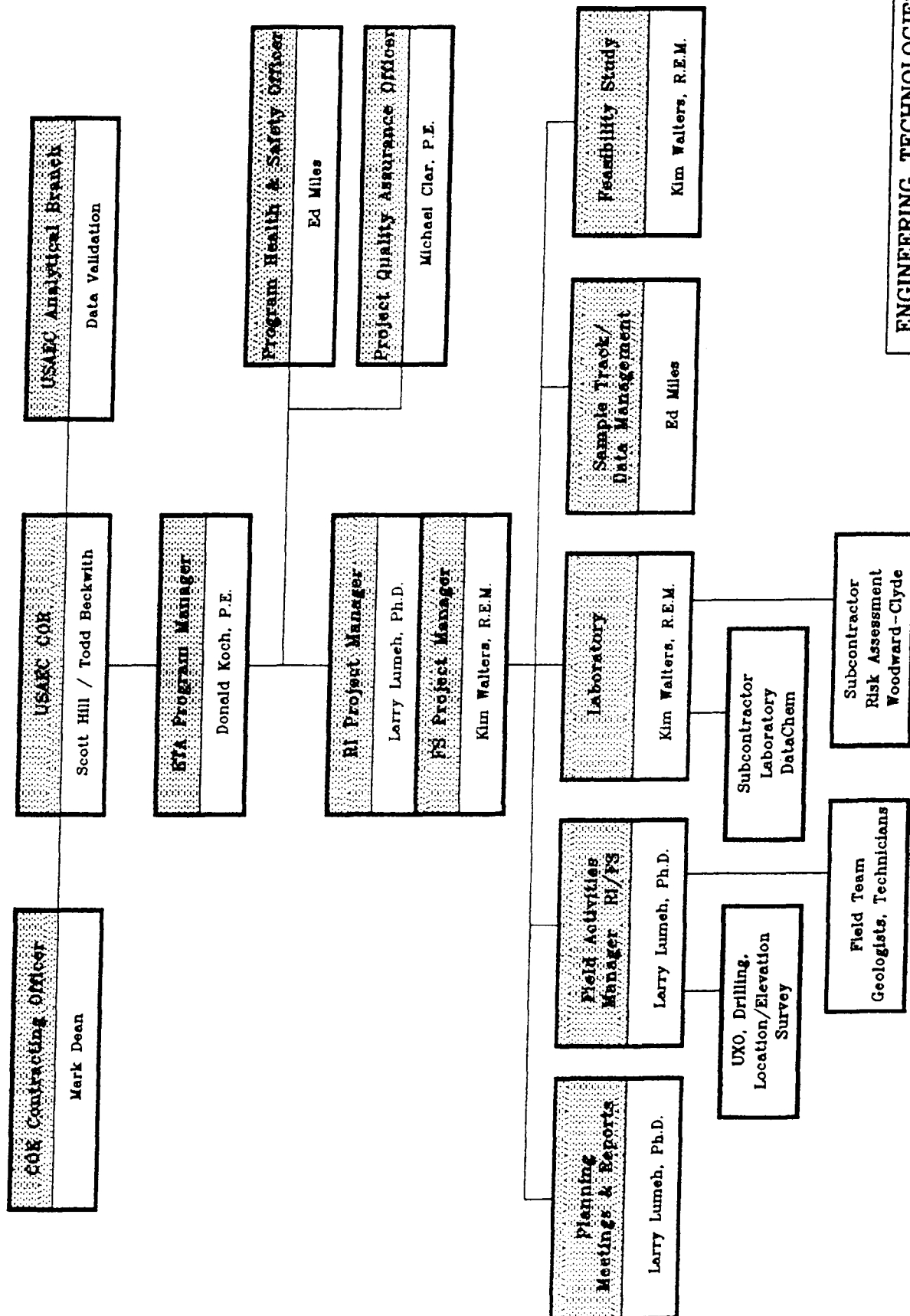
The Feasibility Study Project Manager (FSPM) for this project is Kim Walters, R.E.M. He will be responsible for project staffing and direct management of this portion of the project. He will institute financial and schedule controls and will review and approve all deliverables prior to their submittal to USAEC. He will maintain a liaison with the USAEC Project Officer and the Fort Meade Environmental Office, keeping them informed of technical progress of this phase of the project. He will also manage the completion of all community relations related activities.

#### **2.1.4 Additional Staff**

To assist the above mentioned staff in the successful completion of this project the following staff will perform the following roles:



FIGURE : 3  
DRMO RI / FS ORGANIZATION CHART



**ENGINEERING TECHNOLOGIES  
ASSOCIATES, INC.**  
ENGINEERS • PLANNERS • SURVEYORS  
444 SOUTH COLEMAN DRIVE, SUITE 101  
AUSTIN, TEXAS 78746  
P.O. BOX 99-0001 TEL: 512-441-9999

- |   |  |                                |
|---|--|--------------------------------|
| ● | Field Activities Manager               | Larry Lumeh, Ph.D.             |
| ● | Laboratory Analysis and QA/Data Review | DataChem (subcontractor)       |
| ● | Sample Tracking Database Management    | Kim Walters, R.E.M.            |
| ● | Data Validation                        | Woodward-Clude (subcontractor) |
| ● | Risk Assessment                        | Woodward-Clyde (subcontractor) |

## **2.2 Engineering Technologies Associates, Inc. QA/QC Organization**

In order to ensure that all aspects of QA/QC are followed according to the USAEC QAP and this QCP, the responsibilities to oversee this project have been assigned to the Project QA Officer and the Project Lead Chemist.

### **2.2.1 Project QA Officer**

ETAs Total Quality Management (TQM) Program is directed by Michael Clar, P.E. Mr. Clar is a principal of ETA and Director of the Construction and Remediation Group. He will function as an independent evaluator of ETAs performance during this project and will discuss his findings with the Program Manager and the Project Managers.

The objective of the Project Quality Assurance Officer is to ensure the necessary systems are in place to maintain the maximum level of quality during the lifespan of this project. The particular functions and duties of the Project Quality Officer include:

- Reviewing and approving of QA policies and procedures
- Reporting the adequacy, status and effectiveness of the QA program on a regular basis to the project management
- Maintaining responsibility for documentation of corporate QA records, documents and communications
- Conducting field audits
- Coordinating with the Lead Chemist, as needed, to ensure QC procedures specific to the laboratory and data management are followed and documented

It is advisable that field audits be performed to ensure that all sampling efforts are carried out in accordance with the QA Program.

### **2.2.2 Lead Chemist**

Woodward-Clyde Federal Services (WCFS) will provide the Project Lead Chemist for the project. The Lead Chemist will be responsible for oversight on the project. Specific duties will include the following:

- Maintaining copies of the laboratory documentation, including USAEC-performance demonstrated methods and Quality Assurance Plans
- Providing an external, thereby, independent QA review of DataChem laboratory activities and documentation
- Coordinating with USAEC, ETA and DataChem to ensure that QA objectives appropriate to the project are established and that DataChem personnel are aware of these objectives
- Coordinating with DataChem management and personnel to ensure that QC procedures, appropriate to demonstrate data validity and sufficient to meet QA objectives, are developed and in place
- Ensuring data are properly reviewed by a Woodward-Clyde chemist, including resolving any discrepancies between DataChem and the validator
- Requiring and/or reviewing corrective actions taken in the event of QC failures
- Reporting non-conformance with QC criteria or QA objectives, including an assessment of the impact of the data quality or project objectives, to the Project QA Officer and Project Manager

### **2.3 DataChem Project QA/QC Organization**

The laboratory organization is described in the DataChem Laboratories QA Program Plan which is included as Appendix A of this plan.

### **3.0 QA OBJECTIVES FOR MEASURING DATA IN TERMS OF PRECISION, ACCURACY AND REPRESENTATIVENESS, COMPLETENESS AND COMPARABILITY**

#### **3.1 Introduction**

QA objectives set out the degree of quality necessary in project data for specific project and/or regulatory decisions to be made. Thus the QA objectives developed for this project will ensure that generated data are of the quality necessary for their intended use. These objectives can be expressed in terms of precision, accuracy, representativeness, completeness and comparability.

#### **3.2 QA Objectives for the DRMO Project**

The data collected as part of the RI at the DRMO Yard site must meet the QA objectives as set out in this QCP so that the data can be used to make the appropriate decisions as the project proceeds. So that a standard level of data quality can be achieved on all its projects, USEAC determines what standard analytical methods will be implemented. In all cases possible, USAEC analytical methods will be employed for the analysis of DRMO Yard samples. If no USAEC method exists, standard EPA methods will be used. DataChem, the laboratory subcontracted to perform the analyses of environmental samples, is a USAEC-performance demonstrated laboratory. Their QA Program Plan is included in Appendix A of this document.

USAEC-performance demonstrated methods will be used for the following analyses:

- Target Analyte List (TAL): metals analyzed by inductively coupled argon plasma emission spectroscopy (ICP), graphite furnace and cold vapor atomic absorption spectroscopy (GFAA CV) and cyanide by absorption spectroscopy
- Target Compound List (TCL): volatiles analyzed by purge and trap/gas chromatography/mass spectrometry, PAT/GC/MS
- TCL: semivolatiles analyzed by extraction followed by gas chromatography with mass spectrometry, GC/MS
- Polychlorinated biphenyls (PCBs): analyzed by extraction followed by gas chromatography with electron capture detection, GC/ED
- Explosives: analyzed by high performance liquid chromatography with ultraviolet detection, HPLC
- Sulfide: analyzed by ion chromatography, IC

The US Environmental Protection Agency Contract Laboratory Program (CLP) determines what metals are on the TAL and what volatiles and semivolatiles are on the TCL. The exact analytes included as part of these analyses are included on Table 1.

**TABLE 1: TAL/TCL LIST WITH USAEC CODES, CERTIFIED REPORTING LIMITS AND MCLs**

**USAEC Volatile Organic Compounds**

USAEC ANALYTE	ANALYTE CODE	CERTIFIED REPORTING LIMIT		MCL (mg/L)
		(ug/g) soil	(ug/L) water	
1,1,1-trichloroethane	111TCE	1.0	0.20	0.2
1,1,2-trichloroethane	112TCE	1.0	0.33	0.005
1,1-dichloroethene	11DCE	1.0	0.27	0.007
1,1-dichloroethane	11DCLE	1.0	0.49	NR
1,2-dichloroethene (cis,trans)	12DCE	5.0	0.32	cis-0.07 trans-0.1
1,2-dichloroethane	12DCLE	1.0	0.32	0.005
1,2-dichloropropane	12DCLP	1.0	0.53	0.005
1,3-dichloropropene	13DCPE	4.8	0.20	NR
2-chloroethylvinylether	2CLEVE	3.5	0.50	NR
acetone	ACET	8.0	3.3	NR
bromodichloromethane	BRDCLM	1.0	0.20	0.1*
cis-1,3-dichloropropene	C13DCP	NR		NR
vinyl acetate	C2AVE	NR		NR
vinyl chloride	C2H3CL	12.0	1.8	0.002
chloroethane	C2H5CL	8.0	0.64	NR
benzene	C6H6	1.0	0.10	0.005
carbon tetrachloride	CCL4	1.0	0.31	.005
methylene chloride	CH2CL2	1.0	4.4	0.005
bromomethane	CH3BR	14.0	0.26	NR
chloromethane	CH3CL	1.2	0.96	NR
bromoform	CHBR3	11.0	0.20	0.1*
chloroform	CHCL3	1.0	0.24	0.1*
dichloromethane	CH2CL2	1.0	4.4	0.005
chlorobenzene	CLC6H5	1.0	0.10	NR

carbon disulfide	CS2	NR		NR
dibromochloromethane	DBRCLM	1.0	0.25	0.1
ethylbenzene	ETC6H5	1.0	0.19	0.7
toluene	MEC6H5	1.0	0.10	1
methyl ethyl ketone	MEK	10.0	4.3	NR
methyl isobutyl ketone	MIBK	1.4	0.63	NR
styrene	STYR	NR		.1
trans-1,2-dichloroethene	T12DCE	5.0	3.2	.1
trans-1,3-dichloropropene	T13DCP	4.8	0.20	NR
1,1,2,2-tetrachloroethane	TCLEA	1.5	0.20	NR
tetrachloroethene	TCLEE	1.0	0.16	.005
trichloroethene	TRCLE	1.0	0.23	.005
xylenes, total	TXYLEN            M-Xylene O-Xylene	1.0	0.23	10
		2.0	0.78	
trichlorofluoromethane	TCFM	1.0	0.23	NR
dichlorodifluoromethane	DCDFM	NR		NR
USAEC Semivolatile Organic Compounds				
bromacil	BRMCIL	NR	2.9	NR
1,2,4-trichlorobenzene	124TCB	2.4	0.22	0.07
1,2-dichlorobenzene	12DCLB	1.2	0.042	0.6
1,3-dichlorobenzene	13DCLB	3.4	0.042	0.075
1,4-dichlorobenzene	14DCLB	1.5	0.034	NR
2,4,5-trichlorophenol	245TCP	2.8	0.49	NR
2,4,6-trichlorophenol	246TCP	3.6	0.061	NR
2,4-dichlorophenol	24DCLP	8.4	0.0065	NR
2,4-dimethylphenol	24DMPN	4.4	3	NR
2,4-dinitrophenol	24DNP	176	4.7	NR
2,4-dinitrotoluene	24DNT	5.8	1.4	NR
2,6-dinitrotoluene	26DNT	6.7	0.32	NR
2-chlorophenol	2CLP	14	0.35	NR
2-chloronaphthalene	2CNAP	2.6	0.24	NR
2-methylnaphthalene	2MNAP	1.3	0.032	NR
2-methylphenol/2-cresol/o-cresol	2MP	3.6	0.098	NR
2-nitroaniline            ##	2NANIL	31 (20)	3.1 (20)	NR

2-nitrophenol	2NP	8.2	1.1	NR
3,3-dichlorobenzidine	33DCBD	5	1.6	NR
3,4-dinitrotoluene	34DNT	Non-Target Analyte		NR
3-nitroaniline	3NANIL	15	3.0	NR
3-nitrotoluene	3NT	2.9	0.34	NR
4,6-dinitro-2-cresol/-2-methylphenol ## -4,6-dinitrophenol	46DN2C	50 (5)	0.80	NR
4-bromophenyl phenyl ether	4BRPPE	22	0.041	NR
4-chloroaniline ##	4CANIL	1 (0.5)	0.63 (5)	NR
4-chloro-3-cresol/ 3-methylphenol-4-chlorophenol	4CL3C	8.5	0.93	NR
4-chlorophenyl phenyl ether	4CLPPE	23	0.17	NR
4-methylphenol/4-cresol/p-cresol	4MP	2.8	0.24	NR
4-nitroaniline ##	4NANIL	31 (20)	3.1 (20)	NR
4-nitrophenol	4NP	96	3.3	NR
acenaphthene	ANAPNE	5.8	0.041	NR
acenaphthylene	ANAPYL	5.1	0.033	NR
anthracene	ANTRC	5.2	0.71	NR
bis (2-chloroethoxy) methane	B2CEXM	6.8	0.19	NR
bis (2-chloroisopropyl) ether	B2CIPE	5	0.44	NR
bis (2-chloroethyl) ether	B2CLEE	0.68	0.36	NR
bis (2-ethylhexyl) phthalate	B2EHP	7.7	0.48	NR
benzo [A] anthracene	BAANTR	9.8	0.041	0.0001
benzo [A] pyrene	BAPYR	14	1.2	0.0002
benzo [A] fluoranthene	BBFANT	10	0.31	NR
butylbenzylphthalate	BBZP	28	1.8	0.1
benzoic acid ##	BENZOA	3.1 (2)	3.1 (2)	NR
benzo [G,H,I] perylene	BGHIYP	15	0.18	NR
benzo [K] fluoranthene	BKFANT	10	0.13	0.0002
benzyl alcohol	BZALC	4.0	0.032	NR
chrysene	CHRY	7.4	0.032	0.0002
hexachlorobenzene	CL6BZ	12	0.080	NR
hexachlorocyclopentadiene	CL6CP	53	0.52	0.05
hexachloroethane	CL6ET	8.3	1.8	NR
dibenz [A,H] anthracene	DBAHA	12	0.31	0.0003

dibenzofuran	DBZFUR	5.1	0.38	NR
diethyl phthalate	DEP	5.9	0.24	NR
dimethyl phthalate	DMP	2.2	0.63	NR
di-n-butyl phthalate/dibutyl phthalate	DNBP	33	1.3	NR
di-n-octyl phthalate	DNOP	1.4	0.23	NR
fluoranthene	FANT	24	0.032	NR
fluorene	FLRENE	9.2	0.065	NR
hexachlorobutadiene	HCBD	8.7	0.97	NR
indeno [1,2,3-C,D] pyrene	ICDPYR	21	2.4	0.0004
isopropylamine	IPA	Non-Target Analyte		NR
isophorone	ISOPHR	2.4	0.39	NR
naphthalene	NAP	0.23	0.74	NR
nitrobenzene	NB	3.7	1.8	NR
N-nitroso-di-n-propylamine	NNDNPA	6.8	1.1	NR
N-nitroso-diphenylamine	NNDPA	3.7	0.29	NR
pentachlorophenol ##	PCP	9.1	0.76	0.001
phenanthrene	PHANTR	9.9	0.032	NR
phenol	PHENOL	2.2	0.052	NR
pyrene	PYR	17	0.083	NR

#### USAEC Metals Compounds

silver	AG	10.0	.0803	NR
aluminum	AL	112	11.2	NR
arsenic	AS	117	16.4	0.05
barium	BA	2.82	3.29	2.0
beryllium	BE	1.12	0.427	0.004
calcium	CA	105	25.3	NR
cadmium	CD	6.78	1.2	0.005
cobalt	CO	25.0	2.50	NR
chromium (total)	CR	16.8	1.04	0.1
copper	CU	18.8	2.84	NR
cyanide	CN	5.0	0.25	0.2
iron	FE	77.5	6.66	NR
mercury (inorganic)	HG	0.10	0.05	0.002



potassium	K	1240	131	NR
magnesium	MG	135	10.1	NR
manganese	MN	9.67	9.87	NR
sodium	NA	279	38.7	NR
nickel	NI	32.1	2.74	0.1
lead	PB	43.4	7.44	.005
antimony	SB	60.0	19.6	0.006
selenium	SE	97.1	20.7	0.05
thallium	TL	125	34.3	0.002
vanadium	V	27.6	1.41	NR
zinc	ZN	18.0	2.34	NR

**USAEC Polychlorinated Biphenyl Compounds**

PCB 1016	PCB016	-	0.32	0.0005
PCB 1221	PCB221	-	-	0.0005
PCB 1232	PCB232	-	-	0.0005
PCB 1242	PCB242	-	-	0.0005
PCB 1248	PCB248	-	-	0.0005
PCB 1254	PCB254	-	-	0.0005
PCB 1260	PCB260	0.176	0.0479	0.0005

NR - No Record

\* - cannot exceed 0.1 for all trihalomethanes

PQL- Practical Quantitative Limit

## - Non certified target analyte for this matrix. Standards are analyzed and results are reported as ND at the PQL.

The number in parentheses is the concentration of the standard from the curve in which we can reliably detect the compound.  
Currently, the first number is used as the PQL in IRDMIS, but a more accurate number is the one in parentheses.

For health and safety reasons and to provide real-time data, field screening measurements will be collected and logged. These field measurements include pH, temperature, conductivity and volatile organics. Portable equipment will be used to record these data which are comparable to EPA Level I data. Table 2 shows the data quality objectives for critical measurements in terms of accuracy and completeness for all parameters analyzed for this investigation.

Toxicity Characteristic Leaching Procedure (TCLP) for volatiles, semivolatiles, metals, herbicides and pesticides will be performed using the standard EPA methods shown below with specified QA/QC requirements.

**Table 2: Data Quality Objectives for Critical Measurements: Precision, Accuracy, and Completeness**

Lab/Field QC	Parameters	Matrix	Estimated Accuracy <sup>a</sup>	Estimated Precision <sup>a</sup>	Completeness
Lab USAEC-PD <sup>1</sup>	TCL VOAs	Soil/Sediment	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	TCL SEMI VOAs & bromacil	Soil/Sediment	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	TAL Metals	Soil/Sediment	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	PCBs	Soil/Sediment	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	Sulfide	Soil/Sediment	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	TCL VOAs	Ground Water	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	TCL SEMI VOAs & bromacil	Ground Water	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	TAL Metals	Ground Water	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	Cyanide	Ground water	USAEC	USAEC,RPD 50% <sup>b</sup>	90%
Lab USAEC-PD <sup>1</sup>	Sulfide	Ground Water	USAEC	USAEC,RPD < 50% <sup>b</sup>	90%
Field Non-PD <sup>2</sup>	pH	Ground Water	+/- 0.2 pH units	+/- 0.2 pH units <sup>b</sup>	90%
Field Non-PD <sup>2</sup>	Temperature	Ground Water	+/- 1 C	+/- 1 C <sup>b</sup>	90%
Field Non-PD <sup>2</sup>	Conductivity	Ground Water	+/- 2% scale	+/- 2% scale <sup>b</sup>	90%
Field Non-PD <sup>2</sup>	Turbidity	Ground Water	+/- 2% scale	+/- 2% scale <sup>b</sup>	90%
Lab Non-PD <sup>3</sup>	TCLP VOAs	TCLP Extract	Compound Dependent	Compound Dependent	90%
Lab Non-PD <sup>3</sup>	TCLP Semi-VOAs	TCLP Extract	Compound Dependent	Compound Dependent	90%
Lab Non-PD <sup>3</sup>	TCLP Metals	TCLP Extract	+/- 15%	RPD < 10%	90%
Lab Non-PD <sup>3</sup>	Total Dissolved Solids	Ground Water	+/- 20%	RPD < 30% RPD < 50% <sup>b</sup>	90%

Sources:

1. USAEC, Quality Assurance Program, January 1990
2. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983
3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, January 1990
  - a. For the USAEC-performance demonstrated(PD) methods, the precision and accuracy limits will be based on the historical control chart data of DataChem Laboratories. For the non-performance demonstration methods, the precision will be based on recovery of spikes using USAEC standard soil and water.
  - b. RPD-DQO is for the analysis of field duplicates.

Analyses	Media	EPA Method
TCLP	Soil	Full Suite
Extraction	Leachate	1311
GC/MS Vol.	Leachate	8240
GC/MS Semi-Vol.	Leachate	8270
Pesticides/PCB's	Leachate	8080
Herbicides	Leachate	8150
Metals	Leachate	6010/7471

### 3.2.1 Precision

The degree of mutual agreement among individual measurements of the same parameter using a prescribed condition and a single test procedure is referred to as precision. The results of the duplicate analyses are computed and the absolute relative percent difference (RPD) is calculated using the following formula:

$$\text{RPD} = \frac{(\text{Sample result} - \text{Duplicate result})}{\text{Average result}} \times 100$$

Laboratory precision is evaluated, for the USAEC-performance demonstrated methods, as part of the control chart program. A three-day moving average control chart is maintained for each control analyte by plotting the range of recovery of spiked QC samples; an updated three-day average range of recovery for each compound is plotted on the control chart as part of the daily laboratory control program. Evaluation of the control charts helps monitor variations in the precision of routine analysis and detect trends in observed variations.

### 3.2.2 Accuracy

The difference between individual analytical measurements and the true or expected value of a measured parameter is referred to as accuracy. The actual test result is compared to the theoretical result of 100% recovery and the percent recovery is calculated using the following formula:

$$\% \text{ Recovery} = \frac{(\text{Spiked sample result} - \text{Sample result})}{\text{Spiked Quantity}} \times 100$$

### 3.2.3 Representativeness

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan.

### 3.2.4 Completeness

Data completeness can be quantified during data assessment. It is expected that laboratories should provide data meeting QC acceptance criteria for 95% or more of the requested determinations. It is incumbent for planners to identify any sample types, such as control or background locations which require 100% completeness.

### 3.2.5 Comparability

Comparability is also considered during the work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. For example, the use of EPA-approved methods and procedures ensure comparability with other data from previous or following investigations.

Comparability is also assured through the use of consistent units of measure. For the RI at the DRMO Yard the units in the Table below will be used:

Parameter	Water	Soil
TCL Volatiles	ug/L	ug/g
TCL Semivolatiles	ug/L	ug/g
TCL PCBs	ug/L	ug/g
TAL Metals	ug/L	ug/g
IC Sulfide	ug/L	ug/g
TOC		ug/g
pH	pH units	NA
Temperature	degrees C	NA
Conductivity	umhos/cm <sup>2</sup>	NA

## 4.0 SAMPLE COLLECTION

The design and planning of the sampling program and the specific sample collection and handling procedures will help determine the quality of the collected data. Additionally, activities ancillary to the collection of samples include the following:

- Preparation of sample containers
- Sample preservation
- Sample identification
- Sample handling and shipment
- Chain-of-custody documentation

### 4.1 Sampling for the Remedial Investigation at the DRMO Yard

The sampling program for the RI at the DRMO Yard is described in the Technical Work Plan which has been provided as a separate document. Sampling methodologies will be performed in accordance with the specifications in Section 6.0 of the USATHAMA Quality Assurance Program Manual. Sections of the manual relating to individual guidelines are summarized in the table below:

Guideline	Manual Reference (Section)
Personnel	6.2
Containers	6.3
Volatiles	6.4
Volatile ground water	6.4.1
Volatile soil	6.4.3
Ground water	6.5
Monitor wells	6.5.1
Surface water	6.6
Soils/Sediments	6.7
Sample preservation	6.9

Soil and water samples will be collected and submitted to DataChem Laboratories for chemical analysis during the installation of monitoring wells, the sampling of monitoring wells and the sampling of surface runoff and sediments. The various sampling and data collection procedures are described in the sections below.

Subsurface soil samples collected during the completion of monitoring wells will be collected and all wells will be completed by a Maryland Licensed Well Driller.

#### 4.1.1 Subsurface Soil Sampling

During the installation of the monitoring wells, subsurface soil samples will be collected at five-foot intervals. The wells will be installed in general accordance with USAEC Geotechnical Specifications. Wells will be installed using a truck-mounted drill rig equipped with 6 5/8-inch, hollow-stem augers. Total well completion depth will be approximately 10 - 15 feet below the water table.

Subsurface soil samples will be collected using split-spoon sampling techniques. Drill cuttings and soil samples will be visually inspected by a Geologist and field classified according to the Unified Soil Classification System. A well boring log will be kept in the field which records vertical variations in sample lithology, odor, relative moisture content, texture and other significant features and events. Drill cuttings and soil samples will be monitored whether they exhibit measurable readings or not using a portable photoionization detector (PID). In this manner field personnel can determine the accuracy of PID field measurements.

Two soil samples will be collected from each split spoon sample and containerized in glass jars. One container will be covered with aluminum foil, closed with a lid and will sit for approximately 15 minutes. The lid will then be removed and the foil pierced with a PID to perform a headspace analysis. The second sample corresponding to that exhibiting the highest readings on the PID, or that collected at the water table from each boring, will be submitted to the laboratory for analysis.

All drilling equipment including the drill rig, hollow stem augers, steel casing, drill rods, mud tubs and split spoon samplers will be steam cleaned and rinsed with distilled water immediately prior to the initiation of drilling activities, prior to relocation on site, and prior to leaving the site. The drill rig and associated equipment will be decontaminated in an area designated for this activity by the Base Commander through the USAEC Project Officer.

#### 4.1.2 Ground Water Sampling

Ground water samples will be collected from all the new and existing monitoring wells, as described in the Technical Work Plan, for analysis. For the two newly constructed monitoring wells, sampling will not occur any sooner than 14 days after their installation.

Prior to collecting the samples, the monitoring wells will be purged by removing three casing volumes of ground water. The physical parameters of temperature, pH, and conductivity will be measured in the field and logged in the field notebook. The individual sample containers and lids will be rinsed with the monitoring well water prior to placing the sample inside. Each sample that requires filtering will be collected by attaching an in-line, 0.45 micron disposable filter to the pump outflow. The samples will be preserved according to EPA protocol, packed

on ice and shipped to the laboratory. Additionally, a field duplicate, a trip blank and a rinsate blank will be collected and shipped along with the samples.

#### **4.1.3 Surface Runoff**

If it can be determined that surface runoff may be flowing from the DRMO Yard onto the BRAC land parcel, surface runoff samples will be collected along the ditches north and south of the DRMO Yard. Because these ditches are typically without water, the sampling must occur subsequent to a rain event.

Samples will be collected at approximately one half to two thirds of the water depth using a decontaminated stainless steel discrete bomb sampler. If the amount of surficial flow is not sufficient to collect a sample, a shallow sample collection basin will be established by installing a two-foot length of four-inch slotted PVC well screen into the subsurface approximately 1.5 feet then placing a PVC slip cap over the sampling port.

Prior to using sampling equipment it will be steamed cleaned and rinsed in distilled water. Prior to collecting the sample, the sample container will be rinsed in the surface runoff downstream from the sampling point. Between sampling events the equipment will be rinsed with distilled water. All samples will be preserved and placed in containers according to the appropriate protocols.

#### **4.1.4 Sediment Sampling**

Sediment samples will be collected in conjunction with the surface runoff samples, however, they will be collected after the surface runoff samples have been collected. This will inhibit the collection of sediment in the water samples. Additionally, so as not to disturb the sediments, the sediment sample location will be approached from downstream. Samples will be placed in containers according to the appropriate protocols.

### **4.2 Location and Elevation Survey**

All sampling points will be plotted on a site sampling map. All newly installed monitoring wells and previously installed MW-200 will be surveyed by a licensed surveyor using NAD27 horizontal and vertical control and USAEC procedures.

### **4.3 Investigation-Derived Wastes**

Potentially hazardous wastes will be generated as a result of the investigations involved in this project. These will be containerized and characterization will be performed in order to determine the appropriate disposal requirements. This characterization will involve collecting drum samples and having the samples analyzed for RCRA TCLP.

## **4.4 Sample Containers, Preservation and Handling**

### **4.4.1 Sample Containers**

In order to ensure the quality of field samples, specific care must be given to the containers that will be used to store the samples. Table 3 shows the analysis to be performed, the type of sample containers, the type of preservation necessary and the holding time.

All sample containers will be supplied by DataChem Laboratories. Sample containers used to collect water samples will be triple-rinsed with the water being sampled, according to USAEC requirements, before the addition of preservatives, except for the volatile sample containers. For volatile analysis, the preservative will be added before the sample container is filled; for all other analyses, the sample container will be filled with sample prior to adding the preservative.

### **4.4.2 Sample Preservation and Holding Times**

Preservation is performed to inhibit the degradation of target analytes in field samples during transport and storage. The specific preservation necessary for this project is shown in Table 3. Sample preservatives will be supplied by DataChem Laboratories and will be added to the sample containers at the time of sample collection. After collection all samples will be stored at 4°C and shipped to the lab.

Holding times, which are calculated from the date of sample collection, are also indicated in Table 3.

## **4.5 Field Quality Control Samples**

Field QC samples will be collected as part of this investigation will include field blanks, trip blanks, rinsate blanks, and field duplicates. They will be included at a rate of 1 per lot or 1 per 20 samples, per sampling technique. Table 4 shows the samples to be collected including their associated QC samples.

## **4.6 Sample Handling**

All samples collected as part of the project will be properly maintained in a manner that assures their integrity and representativeness. It is important that the individual custody of each sample be maintained. A sample is in someone's custody if it is one's actual possession, it is one's view, after being in one's possession, it is in one's physical possession and then locked up so that no one can tamper with it, or it is kept in a secured area, restricted to authorized personnel only.



**Table 3: Containers, Preservation, and Holding Times for Analytical Samples**

Analysis	Sample Containers	Preservation	Holding Times
TCL Volatiles - water	Two 40-mL amber glass VOA vials Teflon-lined cap	HCl to pH < 2 Cool, 4°C	14 days
TCL Volatiles - soil	250-mL amber wide-mouth glass jar, Teflon-lined cap	Cool, 4°C	14 days
TCL Semivolatiles & bromacil - water	1-L amber glass jar, Teflon-lined cap	Cool, 4°C	7 days to extraction; 40 days after extraction
TCL Semivolatiles & bromacil - soil	250-mL amber wide-mouth glass jar, Teflon-lined cap	Cool, 4°C	7 days to extraction; 40 days after extraction
PCBs - water	1-L amber glass bottle, Teflon-lined cap	Cool, 4°C	7 days to extraction; 40 days after extraction
PCBs - soil	250-mL amber wide-mouth glass jar, Teflon-lined cap	Cool, 4°C	7 days to extraction; 40 days after extraction
TAL Metals (ICP/GFAA) - water	1-L Polyethylene bottle, Teflon-lined cap	HNO <sub>3</sub> to pH < 2	6 months
TAL Metals (ICP/GFAA) - soil	250-mL amber wide-mouth glass jar, Teflon-lined cap	Cool, 4°C	6 months
Mercury - water	1-L polyethylene bottle, Teflon-lined cap	HNO <sub>3</sub> to pH < 2	28 days
Mercury - soil	250-mL amber wide-mouth glass jar, Teflon-lined cap	Cool, 4°C	28 days

**Table 3: Containers, Preservation, and Holding Times for Analytical Samples  
(continued)**

Analysis	Sample Containers	Preservation	Holding Times
Sulfide - water	250-mL polyethylene bottle	Cool, 4°C	28 days
Sulfide - soil	250-mL amber wide-mouth glass jar	Cool, 4°C	28 days
TCLP Analytes - water	Two 40-mL VOA vials and Two 1-L amber glass bottles, Teflon-lined cap	Cool, 4°C	**
TCLP Analytes - soil	Two 250-mL amber wide-mouth glass jars, Teflon-lined cap	Cool, 4°C	**
Cyanide water soil	1-L glass or polyethylene 1-L glass or polyethylene	NaOH to pH above 12, cool, 4°C	14 days 14 days

\*\* The analytical holding times for the TCLP samples are provided below.

TCLP Analysis	Max. Time: Sampling to TCLP Extraction	Max. Time: TCLP Extraction to Sample Prep.	Max Time: Sample Prep. to Analysis	Max. Total Elapsed Time from Sample Collection
Volatiles	14 days	-	14 days	28 days
Semivolatiles/ Pesticides/PCBs	7 days	7 days	40 days	54 days
Metals	180 days	-	180 days	360 days
Mercury	28 days	-	28 days	56 days

Source: A.D. Little, 1993.

Each sample will be labeled separately, individually wrapped in bubble wrap and the appropriate information from each sample will be placed on the Chain-of-Custody Form prior to being placed in a rigid cooler. The cooler will contain ice and a thermometer will be used to ensure that the cooler temperature is maintained at 4°C. Chain-of-custody forms and packing lists will be placed inside the cooler prior to shipment. All empty cooler space will be filled in with bubblewrap, and the cooler will be sealed with a custody seal. Figure 4 shows an example of the Chain of Custody for that will be used for this project and Figure 5 shows a copy of a Custody Seal.

Table 4 DRMO Analytical Program

AQUEOUS SAMPLES	TCL									
	LOC	FREQ	SVOCs	VOCs	METALS	PCB	PEST	SO2	CYANIDE	Hg
<b>GROUND WATER SAMPLES</b>										
DRMO-Existing Wells	5	1	5	5	5	5	5	0	5	5
DRMO-New Wells	8	1	8	8	8	8	8	0	8	8
DRMO-Potable Water Well	1	1	1	1	1	1	1	0	1	1
Surface Runoff Samples	4	1	4	4	4	4	4	0	4	4
<b>TOTAL AQUEOUS SAMPLES</b>	<b>18</b>	<b>18</b>	<b>18</b>	<b>18</b>	<b>18</b>	<b>18</b>	<b>18</b>	<b>0</b>	<b>18</b>	<b>18</b>
<b>Quality Control Samples</b>										
Rinsate Blank	1	1	1	1	1	1	1	0	1	1
Trip Blank	1	2	0	2	0	0	0	0	0	0
Duplicate	2	1	2	2	2	2	2	0	2	2
<b>TOTAL AQUEOUS QC SAMPLES</b>	<b>3</b>	<b>5</b>	<b>3</b>	<b>3</b>	<b>3</b>	<b>3</b>	<b>3</b>	<b>0</b>	<b>3</b>	<b>3</b>
<b>GRAND TOTAL AQUEOUS SAMPLES</b>	<b>21</b>	<b>23</b>	<b>21</b>	<b>21</b>	<b>21</b>	<b>21</b>	<b>21</b>	<b>0</b>	<b>21</b>	<b>21</b>
<b>SOIL/SOLID SAMPLES</b>										
DRMO-Subsurface	8	1	8	8	8	8	8	8	8	8
Surface Soil	10	1	10	10	10	10	10	10	10	10
Surface Sediment	4	1	4	4	4	4	4	4	4	4
<b>TOTAL SOIL/SOLID SAMPLES</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>	<b>22</b>
Duplicate	3	1	3	3	3	3	3	3	3	3
<b>GRAND TOTAL SOIL SAMPLES</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>25</b>

NOTES:

- LOC- Locations
- FREQ- Frequency
- TCL VOC - Target Compound List Volatile Organics
- TCL SVOC - Target Compound List Semivolatile Organics
- TCLP - Toxicity Characteristic Leachate Procedure
- PCB- Polychlorinated biphenyls
- PEST- Pesticides
- TOC - Total Organic Carbon
- TAL - Target Analyte List

## USAEC Sample Chain of Custody

[illegible]

Copies: White and Yellow copies accompany sample shipments to laboratory. Yellow copy retained by laboratory. White copy to be returned to ERM for files. Pink copy retained by sampler. Gold copy extra as needed.

10.94

#### Figure 4 CHAIN OF CUSTODY EXAMPLE

<b>CUSTODY SEAL</b>	
Person Collecting Sample _____	Sample No. _____
(signature)	
Date Collected _____	Time Collected _____

**Figure 5 CUSTODY SEAL EXAMPLE**

## 5.0 SAMPLE CUSTODY

Sample chain-of-custody procedures will follow the guidelines in Section 7.0 of the USATHAMA Quality Assurance Program Manual. The objective of these procedures is to provide an accurate written record that can be used to trace the possession and handling of a sample from the moment it is collected until it is analyzed. A sample is considered to be in custody of it is: in someone's physical possession; in someone's view; locked up; or kept in a secured area that can be accessed by authorized personnel.

### 5.1 Field Custody Procedures

It is important that only a limited number of field persons be involved in sample collection and handling. Field sampling techniques such as those published by the U.S. Environmental Protection Agency will be followed. Field records will be completed at the time when each sample is collected and will include the following: date and time, sample or log number, source of sample, analyses required, collector's name, and pertinent field data.

Samples collected for chemical analyses during this RI will be assigned a unique sample identification code composed of the Site Location Identity (SLI) and a Unique Sample Code (USC). The USC and SLI are IRDMIS designated codes which allow information about the sample to be directly linked to a particular sample location.

Each sample, prior to being placed into the sample cooler, will be sealed by placing a custody seal around the cap of the individual sample container that would indicate tampering if removed. Samples will be logged onto the Chain of Custody Record once they return from the sampling location and prior to being placed into the transportation cooler. An example of this Chain of Custody Form is shown in Figure 5. Once the transportation cooler is full, all openings into the cooler will be sealed with custody seals. A copy of a Custody seal is shown in Figure 6. All custody transfers between personnel will be documented in the field notebook.

Color photographs will be taken of the individual sample locations and these photographs will be logged in the field notebook. This log will include the location, date and time and name of the person taking the photograph.

The Field Activities Manager will be responsible for properly packaging and dispatching samples to the appropriate laboratory for analysis. The individual transportation coolers will be accompanied by the Chain of Custody Form. All transportation coolers should be packaged to ensure that samples will not break and all openings into the cooler should be sealed with custody seals.

## **5.2 Laboratory Custody Procedures**

For the RI at the DRMO Yard, laboratory custody procedures will begin when the samples are relinquished to DataChem Laboratories. Their internal custody procedures will consist of the following:

Sample receipt and log-in will be accomplished by the designated sample custodian. All information regarding who receives the sample and from whom the sample was sent will be logged into a permanent log book. Internal chain-of-custody prior to sample analysis will be accomplished by securing the sample in a clean, dry refrigerated room. Samples will be distributed to the appropriate personnel for analysis by the sample custodian. During analysis the individual laboratory personnel are responsible for the care and custody of the sample. Once complete, the unused portion of the sample will be returned to the sample custodian. These unused samples will be retained until permission to dispose of the unused portion is received. More specific custody procedures are summarized in the DataChem QA Program Plan included as Appendix A of this QCP.

## 6.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures are necessary for both field and laboratory equipment. All instruments used during field operations will be operated and calibrated according to the manufacturer's guidelines and recommendations. Operation, calibration and maintenance information will be documented in a field notebook.

### 6.1 Field Instrumentation

Instruments will be calibrated according to the manufacturers specifications at the start of each day's usage. All data from the calibration procedures will be documented in the field notebook and retained within the project file. Failure of a field instrument in meeting calibration guidelines will result in a report to the site coordinator and removing the instrument from usage.

For the purpose of this investigation, daily calibration will be accomplished of the following equipment as shown in the Table below.

Field Equipment Item	Calibration Procedure	Frequency
pH Meter	Two point calibration with solutions of pH 7 and pH 10.	Daily
FID	Using demand regulator and lecture bottle	Daily
conductivity meter	Using conductivity solution	Daily
combustible gas indicator	Using span gas	Daily
geiger counter	Via provided radiation emitter	Daily
PID	Using span gas	Daily
Respirable Dust Indicator	Performed by equipment supplier	Upon receipt of equipment

### 6.2 Laboratory Calibration

DataChem Laboratories will be analyzing all samples collected as part of this investigation. Their analytical instrumentation is calibrated according to a calibration program. The calibrations are performed by DataChem Laboratories personnel using reference standards. A detailed description of their calibration program is contained in their Quality Assurance Program Plan which is included in Appendix A.



## 7.0 ANALYTICAL PROCEDURES

### 7.1 Analytical Program

The chemical analysis program for this RI has been developed to determine the extent and degree of contamination at the DRMO Yard. These data will then be used to analyze remedial alternatives and develop a methodology for remediating the contamination. Therefore a specific set of analytes has been determined for selected chemicals at detection limits consistent with USAEC, state and Federal reporting limits.

The analytical methods listed in the Table below will be used for this RI.

**Summary of Analytical Methods**

Analysis	Method Type	USAEC Method Numbers
TCL Volatiles - water	Class 1A	UM21
TCL Volatiles - soil	Class 1A	LM23
TCL Semivolatiles & bromacil - water	Class 1A	UM25
TCL Semivolatiles & bromacil - soil	Class 1A	LM25
Pesticides/PCBs - water	Class 1B	UH20
Pesticides/PCBs - soil	Class 1A	LH17
TAL Metals (ICP) - water	Class 1	SS12
TAL Metals (ICP) - soil	Class 1	JS12
GFAA Metals - water Arsenic/Lead/Selenium/Thallium	Class 1	AX8/SD18/SD25
GFAA Metals - soil Arsenic/Lead/Selenium	Class 1	B9/JD21/JD20
Mercury - water	Class 1	CC8
Mercury - soil	Class 1	Y9
Cyanide - water	Class 1	TF34
Cyanide - soil	Class 1	KF15
Sulfide - soil	Class 1	Modified TY15
Total Organic Carbon (TOC)	Class 1	Modified Lloyd Kahn

### **7.3 Analyst Qualification**

DataChem will supply qualified personnel to perform the tasks of the analytical team. These qualifications will be documented interims of education, experience and training.

### **7.4 Field Analytical Methods**

The analytical methods to be performed in the field include conductivity, pH and temperature.

## **8.0 DATA REDUCTION, VALIDATION, AND REPORTING**

### **8.1 Engineering Technologies Associates, Inc. Data Management**

All project-related information will be effectively managed including map, geotechnical and chemical data. ETA's and the subcontracted laboratory's data management systems will be integrated to achieve an efficient flow of information from the laboratory to ETA and subsequently to the USAEC.

#### **8.1.1 Flow of Map Data into IRDMIS**

The entry of sampling location into the IRDMIS system requires that the map data conform to specific conventions and the coordinate system provided in the USAEC software program "PC IRDMIS" or "PC TOOL". ETA will record the locations of the new sampling locations to ensure proper processing by Potomac Research, Inc. (PRI) and the entry of the associated analytical data.

#### **8.1.2 Flow of Analytical Data into IRDMIS**

WWC will be responsible for the final validation of 25 percent of the analytical data associated with the sampling efforts at Fort Meade. This review is in addition to the checks performed by the subcontracted laboratory. After the laboratory has analyzed the field samples and prepared the IRDMIS data transfer file, data will be submitted to PRI for eventual Level III status. This transfer will be confirmed by the weekly USAEC status report for each lot. WWC's internal tracking system will also ensure that all field samples have had the proper level of analysis performed and will ensure that the laboratory and the USAEC Project Officer is contacted whenever and wherever discrepancies arise.

### **8.2 Data Reduction**

Data reduction occurs by processes that change either the form of expression or the quantity of data values or number of data items. Raw data from quantitative analysis procedures such as Gas Chromatography (GC), Gas Chromatography/Mass Spectrometry (GC/MS), High Performance Liquid Chromatography (HPLC), Inductively Coupled Argon Plasma (ICAP), and Ion Chromatography (IC) generally consist of peak areas (or peak heights) for the analytes of concern, internal standards, and surrogates. This applies to Class 1, 1A, 1B and TPH/GC-FID. These raw data factors will be converted to concentrations by use of calibration curves or relative response factors that relate peak area to the quantity of the analyte introduced into the instrument. For field methods, the calibration procedures are generally less rigorous than those for Class 1, 1A and 1B.

Data will be collected into either computer-based data files or onto hard copy sheets during the analysis process. In reporting results, rounding to the correct number of significant digits will occur only after all calculations and manipulations are complete. For dilutions, the number of

significant digits will be reduced by one. Each analytical method discussed in Section 7 will describe the data reduction procedures for the subcontracted laboratory analysis results. It will also describe the correct procedure for using method blank results.

All uncorrected values less than the certified (performance generated), including no response, will be reported as "less than" the reporting limit. Results of analyses will be entered into IRDMIS via procedures outlined in the IR Data Management User's Guide (USATHAMA, September, 1992). Non-performance demonstrated analytes will be reported using detection limits documented in the appropriate method and will be flagged for data entry into the IRDMIS Non-THAMA Approved Methods (NTAM) database.

### **8.3 Data Validation**

Laboratory review and certification is an integral part of the project QA program and will be performed on 10 percent of all data packages by the laboratory. Data validation is an independent review of data usability that is beyond the review and certification performed by the laboratory. In order to perform the Risk Assessment in EPA Region III, Woodward-Clyde will validate 25 percent of the data in accordance with Region III's modifications to EPA's national functional guidelines for evaluating organic and inorganic analyses.

The following is a brief outline of the data review and validation process:

- Evaluate for completeness of laboratory data;
- Evaluate data with respect to reporting limits;
- Evaluate data with respect to control limits;
- Review holding time data;
- Correlate laboratory data from related laboratory tests;
- Examine chain-of-custody records;
- Compare data on instrument print-outs with data recorded on worksheets or in notebooks;
- Ensure that the same calibration was used for all samples in a single lot;
- Examine chromatographic outputs and documentation if manual integration was performed;
- Compare standard and sample preparation and injection records with instrument output to ensure that each output is associated with the correct sample;
- Examine calibration and tuning results;
- Check calculations on selected samples to ensure correctness;
- Check that GC/MS library searches have been performed for all unknowns, and that the results have been evaluated and recorded;
- Examine all papers and notebooks to ensure that all pages are initialed, dated, and have sufficient explanation for any changes, and that all items are legible; and
- Compare transfer file, record, and group check results with analysis results.

## 8.4 Data Validation Procedures

The subcontracted laboratory performs its own automated QC checks. The results are reviewed by the analyst supervisor and analytical task manager. The data packages containing the computerized reports and all raw data are completed and submitted with the data package to the QA supervisor.

The project QA Coordinator is responsible for reviewing and approving all data packages before the data are submitted to ETA. Data validation involves a thorough review of all data documentation from the raw data to the reported results contained in the lot folders. Data are considered complete only after they are approved by the QA staff. The review process is performed on every batch of data to ensure that all QA checks required by the method are included in the batch.

With the use of the USAEC Data Review Checklist, a thorough data package audit is performed. This includes checking the control charts, method blanks, standard matrix and sample matrix spike recoveries, surrogate recoveries, calibration curves, certified (performance demonstrated) reporting limits, and units. The subcontracted laboratory QA Coordinator or assistant makes an initial judgment on the acceptability of method blank and other data. Analyst's notebook pages, number of samples and sample identifications, dilutions, percent moisture, sample weights, chain-of-custody forms, standard preparation notebooks, and instrument logbooks, are also included in the review. After ensuring that these items are present and complete, the QA staff proceeds to review the raw data for precision, accuracy, and completeness. The raw data are checked against the reported values, and the appropriate calculations are spot checked.

Any discrepancies found are directed to the analytical task manager for verification, clarification, and/or correction, if necessary. Other questions regarding the data transmission file are addressed directly to Data Management. The questions are usually written under the "Comments" section of the USAEC Data Review Checklist or on separate attachments. Once the questions are satisfactorily answered, the QA staff initials and dates the batch and appropriate sections. The batch folder is then returned to Data Management for entry into IRDMIS.

The control charts are reviewed and transmitted to USAEC and ETA by the laboratory QA Supervisor. The control charts are reviewed by the laboratory coordinator, analytical task manager, and QA staff before any data are transmitted to AEC IRDMIS data files.

Three data levels are used to indicate increasing QA and validation performed on the data. Data reviewed by ETA QA staff and subsequently transmitted to USAEC IRDMIS are considered to be Level I data. At USAEC, PRI loads the data into a computer for group and record checks. Errors, if present, are reported to the USAEC COR and chemist. Based on the nature of the error, the data are corrected or rejected. When the data have successfully passed group and record checks, they are elevated to Level II. Level II data become Level III when they are available to users to create reports and graphs, but they cannot be changed by contractors. Generally, only Level III data are available to the USAEC COR. Under unique circumstances,

the COR may request and receive Level I data. Level I data are used for information purposes only. Major decisions and risk assessments are based on Level III data only.

## **8.5 IRDMIS Record and Group Checks**

After each data packet has been reviewed by key individuals and validated by QA and data management staff, the data file from the packet is loaded into the USAEC IRDMIS systems at the subcontracted laboratory and run through the first record check and then the group check. Every data point is checked using these two routines. IRDMIS record check determines the following:

- Whether file names (such as CGW, CSW) and site type (BORE, WELL) combinations are valid.
- Validity of sampling program and technique, and existence or absence of depth measurement.
- Sample date, preparation/extraction date, and analysis date are compared to determine any holding-time violations.
- All test names are verified as valid, and either performance demonstrated or flagged as non-performance demonstrated, at the time of analysis or at present.
- Value compliance with Certified (Performance Demonstrated) Reporting Limit and Upper Certified (Performance Demonstrated) Limit.
- Correct Boolean values, such as ND, LT.
- Correct QC test, mantissa and exponent values, and uncorrected mantissa and exponent values.
- If required, dilution mantissa, exponent, and moisture content inclusion.
- Whether all required flagging codes are included.

IRDMIS group check determines the following:

- That all test names/analytes found in QC are present in all of the samples.
- That all required QC spikes exist, all spiking levels are valid as determined by the methods table, and no aberrations exist in QC or sample data.

Specific criteria for record checks are based on the specific analytical method and on the current performance demonstration status of the laboratory performing the analysis. These criteria are stored in IRDMIS as certifications (performance demonstrations) tables.

If any errors are found in group and record check that are not addressed on the Data Review Checklist by the laboratory analysts, laboratory project coordinator, or the QA Coordinator, the lot is returned to the laboratory project coordinator, so that the problem can be rectified. If changes to the analytical data are required, the lot is then resubmitted for QA review and, after re-validation, it is again processed through IRDMIS to ensure that any errors have been corrected.

After the data in a lot have successfully passed QA validation and IRDMIS record check and group check, a transfer file of the lot is created and sent to USAEC via modem. The data are again run through record and group check by USAEC, and after passing the data checks, are elevated to Level II.

## **8.6 Data Reporting**

The results for samples analyzed for USAEC projects are entered into IRDMIS. Data created using the IRDMIS can then be electronically transmitted to PRI or a diskette together with a hard copy printouts can be submitted.

All the subcontracted laboratory data are entered on a coding form by the analyst, which is verified by the peer checker and group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USAEC software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to PRI. The subcontracted laboratory retains a duplicate diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data are also reviewed by Data Entry for completeness before release.

The subcontracted laboratory subsequently sends data packages to ETA for final review. Subsequent to the final review, all pertinent documentation in appropriately labeled boxes is delivered to USAEC.

A separate spreadsheet will be developed for Risk Assessment calculations. Summary tables of the validated data and associated qualifiers will be included in the reports.

## **9.0 INTERNAL QC CHECKS AND FREQUENCY**

### **9.1 Control Samples**

Control samples are those that are introduced into a batch of environmental samples to function as monitors of the analytical method. All required QC samples will be prepared from standard matrices or actual field samples and proceed through the complete performance demonstrated analytical method. Stock solutions used to spike QC samples will be prepared independently of stocks used for calibration or performance demonstration samples.

### **9.2 Field Control Samples**

Various types of field QC samples are used to check the cleanliness and effectiveness of field handling methods. Field QC samples help indicate whether project data quality objectives have been met by providing quantitative and qualitative measures of precision, accuracy, representativeness, completeness, and comparability parameters. They are analyzed in the laboratory as samples, and their purpose is to assess the sampling and transport procedures as possible sources of sample contamination and document overall sampling and analytical precision. Field staff may add blanks or duplicates if field circumstances are such that they consider normal procedures insufficient to prevent or control sample contamination, or at the direction of the Project Manager. Rigorous documentation of all field QC samples in the site logbooks is mandatory.

Field QC samples and the recommendations for frequency of collection are briefly described below. The specification and number of field QC samples to be collected at the DRMO Yard at Fort Meade are provided in the Technical Work Plan.

#### **9.2.1 Trip Blanks**

Trip blanks are not exposed to field conditions. Results from the analysis of trip blanks are used to assess potential contamination from everything except ambient field conditions. Trip blanks are prepared at the laboratory prior to the sampling event by adding reagent grade water to a 40-ml VOA vial containing two to three drops of concentrated hydrochloric acid; they are shipped with the sample bottles. One trip blank will be used with every shipment of water samples for volatile organic analysis. Each trip blank will be transported to the sampling location, handled in the same manner as a field sample (except the bottlecap is not removed), and returned to the laboratory for analysis without having been opened in the field.

#### **9.2.2 Field Equipment/Rinsate Blanks**

The results of analyzing field equipment/rinsate blanks are used to document that sampling equipment have been properly prepared and cleaned before field use and that cleaning procedures between samples are sufficient to minimize cross-contamination. Rinsate blanks are prepared on-site by passing analyte-free water over sampling equipment; they are analyzed for all



applicable parameters. If a sampling team is familiar with a particular site, it may be possible to predict the areas or samples that are likely to have the highest concentration of contaminants. The equipment blank sample should be collected after a sample is expected to exhibit high concentrations of target analytes.

Rinsate blanks will generally be collected at a frequency of one per day per equipment type used that day. Rinsate blanks will not be collected for sampling activities using dedicated equipment to collect each sample.

### **9.3 Laboratory Control Samples**

QC data are necessary to determine precision and accuracy and to provide quantitative evidence that the method is performing comparably or better than when documented during method development and performance demonstration. Laboratory-based control samples will consist of standards, surrogates, spikes, and blanks. Data generated from control samples included in each lot will be plotted on control charts to monitor day-to-day variations in routine analyses. For this program, the subcontracted laboratory will follow the approach described by the USAEC QA Program for performance demonstrated methods with respect to laboratory control samples. For non-performance demonstrated methods will follow the specific method directives. Generally, a blank, a spike, and a duplicate will be included in each lot of 20 or fewer samples.

The types of laboratory control samples and the minimum acceptable performance for non-performance demonstrated methods for USAEC projects are briefly described below.

#### **9.3.1 Laboratory Blanks**

In addition to field blank samples, three types of blanks that may be analyzed in the laboratory are calibration blanks, method blanks, and reagent blanks. Method blanks and reagent blanks are used to assess laboratory procedures as possible sources of sample contamination. Calibration blanks establish the analytical baseline against which all other blanks are measured.

- Method blanks are laboratory blanks that correspond to the first step in sample preparation and as such, provide a check on contamination resulting from sample preparation and measurement activities. For USAEC-performance demonstrated procedures, method blanks for water and soil samples consist of a standard matrix that is subjected to the entire sample procedure as appropriate for analytical method being utilized. For non-performance demonstrated methods, the method blank is typically an appropriate volume of laboratory water carried through the entire preparation and analysis procedure.
- Reagent/Solvent blanks are closely related to method blanks, but they do not incorporate all sample preparation materials and analytical reagents in one sample. When a method blank reveals significant contamination, one or more reagent blanks may be prepared and analyzed to identify the source of contamination.

- Calibration blanks consist of pure reagent matrix and are used to zero an instrument's response to the level of analytes in the pure reagent matrix. They do not provide a direct indication of the types, sources, or levels of contamination, but they establish the analytical baseline.

#### 9.3.2 Laboratory Duplicates

Laboratory duplicate samples are defined as two sample aliquots taken from the same sample container and analyzed independently. The results of these analyses serve as an indicator of the precision of the method and the sample results. For non-performance demonstrated methods, duplicates will be prepared with the frequency specified in the referenced method.

#### 9.3.3 Calibration Standards

A calibration standard is prepared in the laboratory by dissolving a known amount of a pure compound in an appropriate matrix. The final concentration calculated from the known quantities is the true value of the standard. The results obtained from these standards are used to generate a standard curve and thereby quantify the compound in the environmental sample.

#### 9.3.4 Spike Sample

A sample spike is prepared by adding to an environmental sample or standard matrix (for AEC-performance demonstrated methods; before extraction or digestion), a known amount of pure compound of the same type that is to be analyzed for in the analysis. The spike may also be a surrogate compound for the analyte of interest. These spikes simulate the background and interferences found in the actual samples and provide a mechanism to verify overall method performance. The calculated percent recovery of the spike is taken as a measure of the accuracy of the total analytical method. For USAEC-performance demonstrated methods, between one and three spiked samples, as specified in each method, will be included in each lot. For non-performance demonstrated procedures, spiked samples will be analyzed with the frequency specified in the method.

#### 9.3.5 Internal Standard

An internal standard is prepared by adding a known amount of pure compound to the environmental sample; the compound selected is not one expected to be found in the sample, but is similar in nature to the compound of interest. Internal standards are added to the environmental sample just prior to analysis.

### 9.4 Concentration and Frequency of Control Samples

One method blank shall be included in each analytical lot, regardless of performance demonstration class. A single method blank/spike for GC/MS procedures (Class 1A) serves as

a standard matrix QC blank and spike. The frequency of QA samples is summarized in Table 4. The spiked QC samples described below will be included in each analytical lot:

**Table 5 Frequency of Lab QC Samples for USAEC-Performance Demonstrated Methods**

		QC SAMPLES FREQUENCY/LOT	
		Method Blank	Spikes
1	Metals	1	3
	Explosives	1	3
	Nitrate	1	3
	PCBs (soil)	1	1
	Sulfide	1	3
	Chloride	1	3
1A	VOAs	1*	1
	BNAs	1*	1
1B	PCBs (water)	1	1

\* = Surrogates only

#### 9.4.1 Class 1 Performance Demonstrated Method

- Two independently-prepared spiked standard matrix QC samples shall contain all the control analytes at a concentration near the upper end of the certified (performance demonstrated) range or approximately 10 times certified (performance demonstrated) reporting limit (CRL).
- One spiked standard matrix QC sample prepared at the regulatory action level or approximately two times certified (performance demonstrated) reporting limit.

Control analytes will be specified in USAEC standardized method. For multi-analyte methods, USAEC will designate the required control analytes. Control limits will be initialized for analytes.

Control charts will be maintained for each control analyte.

#### 9.4.2 Class 1A Performance Demonstrated Method (GC/MS only)

- One independently-prepared standard matrix QC sample (method blank/spike), containing all the performance demonstrated surrogate analytes at approximately 10 times certified (performance demonstrated) reporting limit (not to exceed the upper limit of the certified (performance demonstrated) range). For the method blank/spike, surrogate results represent the QC spike, while unspiked, non-surrogate results represent the method blank.
- Every field sample will be spiked with performance demonstrated surrogate analytes at approximately 10 times certified (performance demonstrated) reporting limit. The spike concentration will be the same for all the samples.

Control analytes will be specified in the USAEC standardized method. Additional non-surrogate target analytes may be specified by the USAEC project officer. Control charts will be maintained for each control analyte.

Results of natural matrix surrogate spikes are reported to IRDMIS. Appropriate flagging codes will be used to indicate any problems with surrogate recoveries.

#### 9.4.3 Class 1B Performance Demonstrated Method

- In addition to the method blank, one independently prepared spiked standard matrix QC sample will be included in each sample lot. The spiked standard matrix must contain all the control analytes at a concentration near the upper limit of the certified (performance demonstrated) reporting limit.

Control analytes will be specified in the USAEC standardized method. USAEC will designate the required control analytes for multi-analyte methods.

### 9.5 Data Reporting for Quality Control

#### 9.5.1 Class 1, Class 1A, and Class 1B Performance Demonstrated Methods

Results for each analyte in the spiked QC sample will be determined using the same acceptable calibration curve that is used for analytical samples in the lot. Raw values below the CRL will be reported as "less than" the reporting limit. All certified (performance demonstrated) data will be entered into IRDMIS by personnel trained in the use of IRDMIS.

The results for the method blank and spiked QC samples will be quantified each day of analysis. A new lot of samples will not be introduced into the analytical instrument until the results for QC samples in the previous lot have been calculated, plotted on control charts, and the entire analytical method has been shown to be in control.

Data from the method blank will be reported, usually as "less than" the CRL for each analyte. Any values above the terms of concentration, will be entered into IRDMIS. Data collected from analyses with contaminated blanks will not be used or will be reported flagged.

## 10.0 PERFORMANCE AND SYSTEM AUDITS

Performance audits are a quantitative evaluation of a measurement system and generally consist of evaluation of a laboratory's performance in analyzing performance evaluation samples and blind samples. The subcontracted laboratory has participated in performance audits by USAEC and has also participated in EPA's water pollution and water supply performance evaluation program.

System audits are a qualitative on-site review and evaluation of the components and implementation of USAEC's QA Program (January 1990). They consist of field, laboratory, and project audits that are performed by qualified personnel from the ETA QA or technical staff or from external regulatory agencies.

The Quality Assurance reviews under this sub-task are systematic evaluations of four aspects of the Fort Meade DRMO project: (1) field/geotechnical activities, (2) laboratory documentation. The field Quality Assurance reviews will be undertaken by the ETA Project QA Officer or his designee. The laboratory Quality Assurance reviews will largely be undertaken by our subcontracted laboratory, with QA oversight provided by the ETA Project Manager will also review IRDMIS data files and USAEC data packages from our subcontracted laboratory prior to sending files and packages to USAEC. These reviews will assure that activities and data are implemented in accordance with the Technical Work Plan and the Quality Control Plan and associated Standard Operating Procedures, provided as a separate document. These documents adhere to the requirements specified in the *USATHAMA QA Program*, and the *USATHAMA Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition, and Reports*.

### 10.1 Field Audits

Field audits will be performed on a variety of projects to determine the accuracy of the field sampling, documentation, and measurement systems. A schedule for field audits for the Fort Meade field sampling effort will be determined by the ETA Project Manager or the Project QA Officer, and USAEC.

Field Quality Assurance reviews will be performed on site for one day during field investigation activities. The reviews will be conducted by the Project Quality Assurance Officer or his designee. Through a combination of on-site observations and on-site and off-site review of documentation, the following will be reviewed to ensure conformance with the above referenced documents:

- Field logbooks and forms
- Field chemical/physical analyses including calibration and QC samples
- Containers and sample preservation used for collected samples.
- Sample storage and security
- Sample containers
- Location and elevation survey

- On-site steam cleaning drill rig procedures prior to drilling activities, between each well, and before leaving the site
- "Dig-safe" and UXO screening procedures
- Confinement and containerization of drilling wastes (waste steam cleaning condensates from drill rigs and the PVC pipe used for casings; drilling fluid, if used; surface runoff, and antifreeze if used)
- Drilling activities (water sources used) and well materials (Ottawa sand, bentonite and grout)
- Well development and presample purging techniques
- Depth measuring techniques
- Accurate drawings and notes of the well's location and drilling operations
- Specified numbers and types of soil, ground water, surface water, and sediment samples are collected and sent to the laboratory
- Custody forms, including sample labels and chain-of-custody records

The Field Checklist provided in Appendix W of the USAEC QA Program PAM-11-4, will be used during this audit. External audits may also be performed by a representative of the USAEC Geology and Chemistry Branch.

## **10.2 Laboratory Audits**

A system internal audit by the subcontracted laboratory Project Manager and QA Coordinator (or designees) is made before any new experimental procedures are implemented. Systems audits are also made for critical functions during the sampling and analysis program. The system audit is of a qualitative nature and consists of an on-site review of the laboratory's QA system and physical facilities for sampling, calibration, and measurement. The results of these reviews will be documented in initial and final laboratory visit checklists.

Critical functions will be audited by the QA Coordinator to verify that:

- Standards, procedures, records, charts, floppy disks, and notebooks are properly maintained
- Actual procedures agree with written instructions
- QA records are adequately filed and maintained to assure protection and retrievability

The QA Coordinator or designee will also assess the results of QC sample analyses.

In addition to internal laboratory audits, USAEC and WWCFS on behalf of ETA will perform external audits. Currently, the subcontracted laboratory is audited by USAEC every six months by representatives of the USAEC Geology and Chemistry Branch.

Findings from audits will be documented in a bound notebook and maintained in a Project QA file. Findings will include observations and notations as to whether approved practices are followed. A summary of findings will be distributed to the subcontracted laboratory QA Officer, the Project Manager, Analytical Coordinator, ETA Project Manager and Lead Chemist, and the USAEC.

#### 10.2.1 Data Review

As required by the USAEC QA Plan, ten percent of the data packages will be reviewed by the subcontracted laboratory Quality Assurance Coordinator. This review serves two purposes; it ensures that all required data and documentation are provided in the package and it checks the content for technical and recordkeeping errors. The reviewer's name and date of review will be recorded on the QAC Checklist, any corrective actions required will also be noted. When the corrective action has been completed the QAC will initial and date the original comment. The QAC's signature on the checklist will indicate that the data are considered valid and usable.

ETA's subcontractor, WWC, will validate an additional 25 percent of the data and provide ETA with USAEC data packages and IRDMIS data files and WWC will transfer reviewed files to IRDMIS. The packages will be chosen to cover as broad as possible a range of analyses and matrices. The Project Manager will assess the completeness of the documentation provided, adherence to the performance demonstrated or other published method, adherence to USAEC quality control requirements and EPA Region III validation guidance acceptability of the quality control data. The Project Manager will also provide a technical review of the data and verify at least one calculation for standard preparation and final reported analyte values from the raw data contained in the data packages to the final reported value on IRDMIS. Any discrepancies or omissions will be discussed promptly with the subcontracted laboratory. A copy of the ETA Project Manager's review will be added to the data package.

Any deviations or problems with data packages will be reviewed with the subcontractor and the laboratory, and appropriate corrective actions will be taken as necessary and will be fully documented.

### 10.3 Project Audits

Project audits may also be performed on files containing relevant project documentation. These audits will be triggered by apparent non-conformance to the USAEC QA Program and/or in response to corrective actions. Project files are evaluated against internal document control standard operating procedures (SOP). Project audits may be performed on a random percentage of projects by the Project QA Officer or his designee.



## **11.0 PREVENTIVE MAINTENANCE**

### **11.1 Field Instruments**

All field instruments and equipment used for sample analysis will be serviced and maintained only by qualified personnel. All repairs, adjustments, routine maintenance, and calibrations will be documented in an appropriate logbook or data sheet that will be kept on file at the field equipment warehouse. The instrument maintenance logbooks will clearly document the date, the description of the problems, the corrective action taken, the result, and who performed the work.

### **11.2 Laboratory Equipment**

The subcontracted laboratory maintains maintenance contracts with the major instrument manufacturers for 24-hour, 7-day per week emergency call service. It performs routine maintenance to prevent instrument malfunction and minimize downtime, and to optimize instrument capabilities.

The schedule of preventative or routine maintenance checks are, in general, outlined within the specific equipment's operation manuals and in the analytical procedures performed. The subcontracted laboratory adheres to these schedules, and it is the responsibility of both the project analyst and management to monitor that these checks are completed.

The laboratory maintains an inventory of replacement parts for all analytical instrumentation; this enables analysts to perform routine maintenance and repair of instruments as needed.

## **12.0 PROCEDURES TO ASSESS DATA ACCURACY, PRECISION, AND COMPLETENESS**

This section describes the statistical analysis of data obtained during analysis of FGGM samples by USAEC-performance demonstrated methods. The calculations described in this section are contained in computer software developed by the USAEC.

The statistical calculations compare the measured concentration of standards in spiked samples with the known spiked concentrations of these target analytes. The measured concentrations are determined from calibration curves constructed according to the standardized method. Recovery factors will not be used to correct measured concentrations during analysis of the performance demonstration data. These calculations must be performed for each target analyte in a method.

### **12.1 Lack of Fit (LOF) and Zero Intercept (ZI) Tests**

All data must be collected during periods when instrumental calibration was in control (i.e., within plus or minus 10 percent of the mean response for inorganics analyses in surface/ground waters and within plus or minus 25 percent of the mean response for all other analyses). Data obtained from valid methods using properly calibrated instruments are expected to be linear and have a zero intercept, when measured concentrations are compared to the target concentrations. This relationship must be tested because calculation of the CRL assumes that a linear relationship exists.

Data obtained during performance demonstration analyses shall be first examined for any outliers before being tested for linearity using the LOF and ZI tests. In the absence or replacement of an outlier, data from each of the performance demonstration analyses shall be pooled and tested for LOF.

### **12.2 Certified (Performance Demonstrated) Reporting Limit (CRL)**

Before any analytical system is employed in a survey, sufficient spikes and blanks will be run to statistically establish the lowest sample concentration to be reported. This concentration is the CRL. For USAEC projects, CRLs shall be determined by using the AEC program with 95 percent confidence limits. This CRL is associated with the entire method and reflects all sample preparation and measurement steps.

The CRL is derived from the following assumptions:

- The relationship between the measured concentration and target concentration is linear
- The variance about the least squares linear regression line is homogeneous over the tested concentration range
- Measured concentrations for a given target concentration are normally distributed

Based on these assumptions, the least squares linear regression line, of the form indicated in Equation 1, can be determined. The performance demonstration performance data (X, Y paired data) are used to determine the slope and Y-intercept of the least squares regression line according to the formulae provided below in Equations 2 and 3; these equations assume that errors occur only in the measured concentration.

$$Y = Y_o + bX \quad \text{Equation (1)}$$

where:

Y = found concentration;  
Y<sub>o</sub> = Y axis (found concentration) intercept;  
b = slope of the line; and  
X = target concentration.

$$\text{slope} = b = \frac{N \sum X_i Y_i - \sum X_i \sum Y_i}{N \sum X_i^2 - (\sum X_i)^2} \quad \text{Equation (2)}$$

where:

N = number of data points;  
X<sub>i</sub> = the i-th target concentration; and  
Y<sub>i</sub> = the i-th found concentration

$$Y \text{ axis intercept} = Y_o = \frac{\sum Y_i - b \sum X_i}{N} \quad \text{Equation (3)}$$

where:

b = slope of the least squares linear regression line from Equation 2.

The equations for the upper confidence limit (Equation 4) and the lower confidence limit (Equation 5) about the regression line are provided below:

$$Y = Y_o + S_{YX} X \pm \left[ \frac{1}{N} + \frac{(X_i - \bar{X})^2}{\sum (X_i - \bar{X})^2} \right]^{1/2} \quad \text{Equation (4)}$$

$$Y = Y_o + bX \pm S_{YX} X \left[ \frac{1}{N} + \frac{(X_i - \bar{X})^2}{\sum (X_i - \bar{X})^2} \right]^{1/2} \quad \text{Equation (5)}$$

where:

$$= S_y \times \left[ \frac{\sum (Y_i - [\bar{Y} + b(X_i - \bar{X})])^2}{N - 2} \right]^{1/2} \quad \text{Equation (6)}$$

$Y_o$  = calculated Y axis intercept;  
 $t$  = Student's t-test for 2-tailed  $P = 0.10$  and  $N - 2$  degrees of freedom;  
 $X$  = the average of all target concentrations; and  
 $Y$  = the average of all found concentrations.

The calculated reporting limit,  $X_d$ , is the value of  $X$  corresponding to a point on the lower confidence limit curve where the value of  $Y$  equals the value of  $Y$  on the upper confidence limit curve at  $X = 0$ . An example of the statistical analysis of reporting limit using the AEC computer software is shown in the USAEC QA Program manual (January 1990).

The calculated reporting limit will be reported as the CRL of the method, provided that at least one of the tested concentrations is at or below the calculated reporting limit. Otherwise, the lowest tested concentration is the minimum level that can be reported as the CRL. The CRL will not be less than the lowest tested concentration.

The data provide an optimistic estimate of the method reporting limit because interferences found in natural samples will be absent. The highest tested concentration will represent the upper limit of reportable data. All sample measurements must be performed within the tested range. A calculated reporting limit higher than the highest target concentration indicates that either an invalid range was chosen or the method is not suitable for analysis of that compound.

### 12.3 Method Performance Demonstration Accuracy

As calculated according to section 12.2, the slope,  $b$ , of the least squares linear regression line of a plot of observed versus target concentrations is a measure of the accuracy of the method. A slope (accuracy) of "plus one" (1.00) indicates 100 percent recovery over the complete analytical method and tested range. Failure to consider the intercept, if it is significantly different from zero, could result in an erroneous estimate of the accuracy. If the intercept is significantly different from zero, then there is a need to investigate whether the blank was correctly applied or if there is some other systematic error in the system. At no time should the laboratory continue until this is investigated. Experimental values may deviate from this expected value. The performance demonstrated data will provide an optimistic estimate of the method accuracy because interferences found in natural samples will be absent. The accuracy estimate for the complete performance demonstration data set is incorporated into the Aec IRDMIS. The slope for the complete data set will be used to indicate the accuracy of the method.

### 12.4 Method Performance Demonstration Standard Deviation

For all method performance demonstration, the standard deviation,  $s$ , will be calculated at each target concentration according to Equation 7. The standard deviation provides an indication of the precision of the analysis. This calculation is performed by the USAEC software.

**Equation (7)**

$$\text{Standard deviation} = S = \left[ \frac{\sum Y_i^2 - \frac{(\sum Y)^2}{N}}{N - 1} \right]^{1/2}$$

where:

$Y_i$  = the measured concentration; and  
 $N$  = total number of  $Y$  values at each target concentration.

### 12.5 Method Performance Demonstration Percent Inaccuracy

For all method performance demonstration, the percent inaccuracy will be calculated at each target concentration according to Equation 8. This calculation is performed by the USAEC software.

$$\text{Percent inaccuracy} = \frac{\bar{Y} - X}{X} (100) \quad \text{Equation (8)}$$

where:

$X$  = target concentration; and  
 $Y$  = average measured concentration at the target concentration.

### 12.6 Method Performance Demonstration Percent Imprecision

For all method performance demonstration, the percent imprecision will be calculated at each target concentration according to Equation 9. This calculation is performed by the USAEC software.

$$\text{Percent imprecision} = \frac{S}{\bar{Y}} (100) \quad \text{Equation (9)}$$

where:

$S$  = standard deviation; and  
 $Y$  = average measured concentration at the particular target concentration.

where:

S = standard deviation; and  
Y = average measured concentration at the particular target concentration.

## 12.7 Data Moving-Average Accuracy and Precision

Moving-average control charts will be maintained for the specified surrogates in the spiked standard matrix sample (Class 1A). The X - R three-point moving-average control chart will be constructed for each control analyte as follows:

- Use percent recovery to allow for minor variations in spiking concentrations
- The first plotted point is the average of the first three recoveries (from performance demonstration, at concentrations nearest the spiking level)
- Subsequent points are obtained by averaging the three most recent individual recovery values (outliers excluded from calculation but not from plot)
- The range for each point is the difference between the highest and lowest value for each group of three values
- The central line, upper warning limit (UWL), upper control limit (UCL), lower warning limit (LWL), and lower control limit (LCL) for the control charts are calculated using the following formulas:

$$\text{Average} = \bar{\bar{X}} = \frac{\sum \bar{X}}{K} \quad \text{Equation (10)}$$

$$\text{Range } \bar{R} = \frac{\sum R}{K} \quad \text{Equation (11)}$$

where:

X = between-group average of the average recovery of the three points (within group);  
X = average within-group recovery for the three points;  
R = within-group difference between recoveries for data pairs; and  
K = cumulative number of pairs in the database.

Upper Warning Limit (UWL) on Average:

$$UWL_x = \bar{\bar{X}} + 0.682 \bar{R}$$

Lower Control Limit (LCL) on Average:

$$LCL_x = \bar{\bar{X}} + 1.023 \bar{R}$$

Upper Warning Limit (UWL) on Range:

$$UWL_R = 2.050 \bar{R}$$

Upper Control Limit (UCL) on Range:

$$UCL_R = 2.575 \bar{R}$$

Lower Warning Limit (LWL) on Range:

$$LWL_R = 0$$

Lower Control Limit (LCL) on Range:

$$LCL_R = 0$$

All data will be plotted, regardless of whether the lot is in control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual measurement value will be tested as an outlier using Dixon's test at the 98 percent confidence level (USATHAMA QA Program manual (January 1990), Appendix K). If the datum is not classified as an outlier by the test, the point will be included in updating the control chart limits. If an individual measurement is classified as an outlier, it will be used in calculating the three-point moving average for plotting purposes only; the measurement is then excluded from calculations based on the three most recent acceptable individual points that are used to determine moving-average and the control chart limits. Method control will be judged according to the criteria in Section 8.0.

After the first control chart points, control limits will be recalculated using only in-control data points. Any points falling outside of the control limits (UCL or LCL) will be dropped from the calculations (but left on the charts) and the control limits recalculated using only points between the UCL and LCL. Charts will then be updated with the newly calculated control limits and all points plotted.

Lots associated with points outside of the new control limits may require resampling and/or reanalysis as determined by the USAEC COR on a case-by-case basis. These limits will then be used to control analysis of the next 20 lots. The control charts are now the outlier test, although individual measurements will continue to be tested as outliers if they appear not be representative of the data set. A maximum of the 40 most recent lots will be used to recalculate control limits for 60 or more lots (40-point slide).

When, as a result of audits or QC sample analysis, sampling or analysis systems are shown to be unsatisfactory, a corrective action shall be implemented. The Laboratory QA Coordinator will be notified and the necessary corrective action taken.

## 12.8 Control Charts

For Class 1, Class 1A, and Class 1B performance demonstrated methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts are not adjusted for accuracy. Control charts are not used with Class 2 performance demonstrated methods.

Control charts include the analyte, method number, the subcontracted laboratory's code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USAEC-supplied computer control chart program:

- Single-Day X-Bar Control Chart (High Spike Concentration)
- Single-Day Range Control Chart (High Spike Concentration)
- Three-Day X-Bar Control Chart (Low Spike Concentration)
- Three-Day Range Control Chart (Low Spike Concentration)

In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the X-axis
- Percent recovery (for X-bar control charts), or range (for R control charts) along the Y-axis
- Upper control limit (UCL)
- Upper warning limit (UWL)
- Mean
- Lower warning limit (LWL), on X-bar charts
- Lower control limit (LCL), on X-bar charts

For some analytes specified by USAEC, warning limits on X-bar charts are deleted.

### 12.8.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of performance demonstration data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are



accepted, based upon the recoveries established during performance demonstration and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and are not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98 percent confidence level. If the datum is not classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. charts are then updated with newly calculated control limits and all points plotted.

#### **12.8.2 Three-Point Moving Average**

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

#### **12.9 Out-of-Control Conditions**

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each data of analysis. Processing of additional lots will not occur until the results of the previous lots have

been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: a value outside the control limits or classified as outlier by statistical test; a series of seven successive points on the same side of the mean; a series of five successive points going in the same direction; a cyclical pattern of control values; or two consecutive points between the UWL and UCL or the LWL and LCL.

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in-control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to two-thirds of the control analytes meet control criteria. Of the remaining control analytes (less than one-third possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than two-thirds of the control analytes are classified as in-control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also Section 10.0) Field sample data effected by the situation are evaluated and reanalyzed as necessary.

When a method is determined to be out-of-control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in-control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

#### **12.10 Non-AEC Methods**

For non-USAEC methods, including laboratory test for Total Dissolved Solids (TDS) and Total Petroleum Hydrocarbons (TPHC) and field tests for pH, temperature, conductivity, turbidity, and total volatile organics (by photoionization detection), the QC samples and procedures for assessing data precision and accuracy are provided in the referenced method or Standard Operating Procedure.

#### **12.11 Completeness**

Completeness is a measure of the amount of usable data obtained from a measurement system compared to the total amount expected to be obtained. It is calculated as follows:

### 13.0 CORRECTIVE ACTIONS

When, as a result of staff observations, audits or QC sample analysis, sampling or analysis systems are shown to be unsatisfactory, corrective action will be implemented. Staff and management at ETA and/or the subcontracted laboratory may be involved in the corrective action. If previously reported data are affected by the situation requiring correction or if the corrective action will impact the project budget or schedule, the action will directly involve the Project Manager, the USAEC COR, and the USAEC Quality Assurance Chemist. Corrective actions are of two kinds:

- Immediate - to correct or repair nonconforming equipment and systems. The need for such an action will most frequently be identified by the field technician or analyst actually doing the work.
- Long-term - to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include:
  - Staff training in technical skills or in implementing the QA Program
  - Rescheduling of laboratory and/or sampling routines to ensure analysis within allowed holding times
  - Identifying vendors to supply reagents of sufficient purity for field work
  - Revising QA system or replacing personnel
  - Personnel reassignment
  - Field instrumentation replacement

For either immediate or long-term corrective actions, the steps comprising a closed-loop corrective action system are as follows:

- Define the problem
- Assign responsibility for investigating the problem
- Investigate and determine the cause of the problem
- Determine a corrective action to eliminate the problem
- Assign and accept responsibility for implementing the corrective action
- Establish effectiveness of the corrective action and implement the correction
- Verify that the corrective action has eliminated the problem

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, corrective action employed, and verification that the problem has been eliminated will be documented.

In addition, if the corrective action results in the preparation of a new standard or calibration solution(s), then a comparison of the new versus the old solution will be performed and the results supplied with the weekly QC submittal as verification that the problem has been eliminated.

### **13.1 Field Situations**

Deviations from quality in field operation that require corrective action in the field will be identified by field audits as described in Section 10.0 and by other more immediate occurrences, such as equipment malfunction and on-site observations by the field supervisor. Once the problem has been identified, prompt and appropriate action will be taken by the field staff, Project Manager or field supervisor to correct the situation. After a corrective action has been implemented, its effectiveness will be verified and documented in the site log. If the action does not resolve the problem, appropriate personnel will be assigned by the Program Manager to investigate and effectively remediate the problem.

Documentation of all corrective action is required. Immediate corrective actions taken in the field will be documented in the field logbooks and approved by the field supervisor or Task Manager. Corrective actions that result in deviations from the Technical Work Plan or Project QCP will also be documented in a memorandum to the ETA Project Manager and QA Officer. They will ensure appropriate changes are incorporated into the final report. Corrective actions initiated as a result of a field audit must be documented in a memorandum from the Program QA Officer to the Project Manager.

### **13.2 Laboratory Situations**

If weaknesses or problems are uncovered during system or performance audits or QC sample analysis, corrective action will be initiated immediately. The subcontracted laboratory Project Manager, Analytical Coordinator, QA Coordinator, and analyst must be involved in the corrective action. If previously reported data or project schedule or budget will be affected, then the corrective actions planned will be directly reported to the subcontracted laboratory's Project Manager, and ETA Project Manager. Corrective actions may also be initiated by the analyst as required from daily review of control charts.

Corrective action might include, but not necessarily be limited to: recalibration of instruments using freshly prepared calibration standards; replacement of lots of solvent or other reagents that give unacceptable values; instrument repair, additional training of laboratory personnel in correct implementation of sample preparation and analysis methods; and reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements.

## 14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

### 14.1 Laboratory Reports

Each daily report generated has a QA section associated with the text. Any matrix characteristics or other physical parameters are noted. The laboratory must confirm that all characteristics indicated by field investigation team match the sample being analyzed by the laboratory. Any discrepancies cause the analysis sequence to be halted.

Normal submissions to the USAEC Geology and Chemistry Branch include the IRDMIS submissions (Section 8.0) and the results of QC activities. During those periods when analyses are being conducted, all tabular QC charts, as described in Section 12.0, must be submitted to the USAEC Geology and Chemistry Branch and ETA on a weekly basis. The QC report must be provided to the USAEC Geology and Chemistry Branch and ETA no later than five working days after analyses for a week are completed. Analysis data shall be defined by the day the analytical instrument was run. All points that indicate an out-of-control situation must be evaluated and explained. Any corrective measures and reanalysis of samples must be fully explained and documented, including procedural changes to prevent recurrence. Printouts generated from control chart software programs provided by USAEC shall be utilized, when available. A checklist included with each control chart submission is shown in Appendix Q of the USAEC QA Program, January 1990.

As an appendix to the project final report, the QAC, in coordination with the Analytical Task Manager and the Project Manager, will provide tabulation of all QC sample data, as well as specific observations delineating the control effectiveness for each analytical method. These observations will include the following:

- QC samples in each lot and how analytical results were combined to prepare control charts
- Spike levels and rationale for choosing those levels
- Possible effects on environmental sample results of detected concentrations in method blanks
- Unique matrix characteristics of environmental samples

If any time during the analytical effort a process was not in control, a discussion will be submitted on:

- Rationale for judging a point as in control, if it appears to satisfy an out-of-control criterion listed in Section 9.0
- Investigation of the out-of-control situation
- Actions taken to bring the process back into control
- Actions taken to ensure that the out-of-control situation did not recur
- Disposition of data acquired while the process was out-of-control

## **14.2 Program QA Officer and Lead Chemist Reports**

The ETA Project Manager will routinely generate reports to keep the Program and Task managers informed of the QA/QC activities during the course of the RI. These reports will be verbal or in the form of a memorandum and will address any findings encountered during their audits and reviews.

**APPENDIX A**

**DATACHEM QUALITY ASSURANCE PLAN**

**QUALITY ASSURANCE PROGRAM PLAN  
for  
U.S. ARMY ENVIRONMENTAL CENTER  
REVISION CONTROL LOG**

**RECORD OF MAJOR REVISIONS**

Revision No.	Revision Date	Affected Section Page		Description of Change
5	7/21/93	ALL	ALL	Document brought under Document Control.

**RECORD OF MINOR REVISIONS**

Revision No.	Revision Date	Affected Section Page		Description of Change
5	7/21/93	4.3.3	9	Updated from ASTM Type I grade water to ASTM Type II grade water.
5	8/12/93	ALL	ALL	Updated Agency references from USATHAMA to USAEC.



## TABLE OF CONTENTS

<b>1.0</b>	<b>DOCUMENT IDENTIFICATION.....</b>	<b>1</b>
<b>2.0</b>	<b>INTRODUCTION.....</b>	<b>2</b>
<b>3.0</b>	<b>ORGANIZATION AND RESPONSIBILITIES.....</b>	<b>3</b>
3.1	Introduction.....	3
3.2	Laboratory Director.....	3
3.3	Project Manager.....	3
3.4	Analytical Task Manager.....	3
3.5	Quality Assurance Coordinator.....	4
<b>4.0</b>	<b>CERTIFICATION.....</b>	<b>6</b>
4.1	Laboratory Certification.....	6
4.2	Analytical Methods.....	6
4.3	Method Certification.....	8
4.4	Analyst Training.....	10
<b>5.0</b>	<b>SAMPLE HANDLING AND ANALYSIS.....</b>	<b>15</b>
5.1	Sample Management.....	15
5.2	Chain-of-Custody.....	16
5.3	Sample Handling Procedures.....	17
5.4	Toxicity Characteristic Leaching Procedure.....	17
5.5	Holding Times.....	17
5.6	Sample Analysis.....	18
5.7	Data Handling.....	22
<b>6.0</b>	<b>ANALYTICAL SYSTEM CONTROLS.....</b>	<b>24</b>
6.1	Sample Control.....	24
6.2	Document Control.....	24
6.3	Quality Control Samples.....	24
6.4	Control Charts.....	25
6.5	Out-of-Control Conditions.....	27
<b>7.0</b>	<b>PREVENTATIVE MAINTENANCE.....</b>	<b>29</b>
<b>8.0</b>	<b>RECORDKEEPING.....</b>	<b>30</b>
8.1	Laboratory Notebooks.....	30
8.2	Logbooks.....	30
8.3	Hard-Copy Output.....	31
8.4	Data Package Preparation.....	31
<b>9.0</b>	<b>AUDITS.....</b>	<b>32</b>
<b>10.0</b>	<b>CORRECTIVE ACTION.....</b>	<b>33</b>
<b>11.0</b>	<b>QUALITY CONTROL REPORTS.....</b>	<b>34</b>

## 1.0 DOCUMENT IDENTIFICATION

**Document Title:** Quality Assurance Program Plan  
for the U.S Army Environmental Center

**Document Control Number:** QA-3/87

**Organization:** DataChem Laboratories (DCL)  
960 W. LeVoy Dr.  
Salt Lake City, Utah 84123

**Director:** James H. Nelson, Ph.D.  
Phone: 801-266-7700

**Quality Assurance:** Lance M. Eggenberger, M.S.  
Phone: 801-266-7700

## **2.0 INTRODUCTION**

This document is the DCL Quality Assurance/Quality Control Plan, prepared in compliance with the requirements of the U.S. Army Environmental Center (USAEC, formerly USATHAMA) with analytical laboratory services in support of the implementation of various installation restoration programs. This plan adheres to, and is an implementation of, the USATHAMA QA Program, January 1990, First Edition.

DCL is committed, in strictly following this plan, to provide to USAEC analytical data that are of a quality that may be used in litigation. All deviations from this plan or the USATHAMA QA Program will be submitted to USAEC for approval prior to implementation in the laboratory. Such deviations will be properly and fully documented.

DCL has conducted analyses for USAEC since 1984 under the 1982 USATHAMA QA Program, the Second Edition (March 1987) of the 1985 USATHAMA QA Program, and the January 1990 USATHAMA QA Program, First Edition.

## 3.0 ORGANIZATION AND RESPONSIBILITIES

### 3.1 Introduction

Ultimate responsibility for the conduct of all projects, and approval for the implementation of all programs at DCL resides with the Laboratory Director, Dr. James H. Nelson. Functional responsibility for the analytical work is delegated to the Project Manager, Mr. David W. Gayer; to the Analytical Task Managers, Mr. A. Brent Torgensen, and Mr. Richard Wade; and to the Quality Assurance Coordinator, Mr. Ronald H. Marsden.

### 3.2 Laboratory Director

The Laboratory Director is responsible to assure that DCL resources are adequately allocated to the project and that sufficient staffing and equipment are provided. He oversees and supports the Quality Assurance Coordinator.

### 3.3 Project Manager

The Project Manager has the responsibility of communication with the USAEC Program Contract Officer and oversees and supports the Analytical Task Managers in development, implementation, and operation of the analytical program organization. He is directly responsible for the interpretation of the provisions of the contract for DCL. The Project Manager is also responsible to assure that QA/QC recommendations and corrective actions are implemented.

The Project Manager is authorized to conduct official discussions with the Program Contract Officer concerning the original contractual agreement and delivery orders, and any subsequent modifications to the contractual agreement and/or delivery orders. Laboratory personnel matters are decided in concert with the Analytical Task Manager and appropriate Section Managers.

### 3.4 Analytical Task Manager

The Analytical Task Manager has the responsibility of implementing the USATHAMA 1990 QA Plan, and for coordinating the sample analysis flow in the laboratory. This will be achieved through the following:

1. Assuring the provision of sufficient equipment, laboratory space, resources, personnel, and quality reagents and materials to properly conduct the required analyses;
2. Supporting the Quality Assurance Coordinator;

3. Submitting documented analytical methods and laboratory certification data to the USAEC Project Officer prior to the analysis of field samples;
4. Ensuring that all provisions of the approved Project Quality Control Plan are fully implemented in the laboratory;
5. Ensuring the implementation of corrective action for any QA/QC deficiencies.

The Analytical Task Manager has the authority to suspend analytical work for quality control problems and to implement corrective actions recommended by the Quality Assurance Coordinator. He also has authority to accept or reject increases in the delivery rate of samples, within the bounds set by the contract. He confers with section managers and the Project Manager on personnel matters when they impact on the project.

### 3.5 Quality Assurance Coordinator

The Quality Assurance Coordinator (QAC) has the responsibility of establishing, overseeing, and auditing specific procedures for documenting, controlling, and validating analytical data quality. This is accomplished, in part, through the following:

1. Monitoring the QA and QC activities of the laboratory to ensure conformance with authorized policies, procedures, and good laboratory practices, and recommending improvements as necessary;
2. Informing the Project Manager and/or the Analytical Task Manager of noncompliance with the approved QA Program;
3. Requesting standard analytical reference materials from USAEC;
4. Ensuring that all records, logs, standard operating procedures, project plans and analytical results are maintained in a retrievable fashion;
5. Ensuring that standard operating procedures and project QA/QC plans are distributed to all appropriate laboratory personnel;
6. In consultation with the analysts and the Analytical Task Manager, establishing appropriate analytical lot size, including the correct QC samples;
7. Establishing the correct procedures and criteria for evaluating whether analytical performance is acceptable and in-control;
8. Ensuring that samples are received and logged properly, including lot sizing, introduction of required QC samples, and numbering of field samples and control samples;
9. Reviewing all laboratory data before those data are released, verifying that data were collected properly under an in-control analytical system;

10. Ensuring that the DCL quality control chemist, or appropriate analysts, are properly preparing QC samples;
11. Maintaining quality control charts, ensuring timely distribution of such charts, documenting corrective actions, and ensuring that analysts implement and document corrective actions as they become necessary;
12. Ensuring that sample logs, instrumentation logs, and all QC documents are properly maintained, including frequency of entries;
13. Discussing control chart results with the Analytical Task Manager and submitting updated, current charts to the USAEC Project Officer on a weekly basis, or as required by USAEC;
14. Maintaining an awareness of the entire laboratory operation to detect conditions which might jeopardize controls of the various analytical systems;
15. As directed by USAEC, auditing sampling documentation and procedures to ensure proper labeling, handling, transportation, and storage.

The Quality Assurance Coordinator has the authority to:

1. Approve all analytical reports;
2. Reject analytical data which does not meet applicable quality control criteria;
3. Require re-performance of sample analyses which are determined to be out-of-control;
4. Evaluate data and determine apparent long-term trends which may require corrective action;
5. Suspend analytical work, when necessary, to assure corrective actions are taken and that an analysis is again in control.

The Quality Assurance Coordinator also attends and participates in conferences for discussion of quality control and quality assurance problems and procedures.

## 4.0 CERTIFICATION

### 4.1 Laboratory Certification

DCL, as a laboratory, rather than as individual analysts, certifies as proficient in conducting analyses for USAEC. Each member of the organization has the education and training necessary to enable that individual to perform assigned functions. A personnel training file is maintained for each individual. Each individual updates the training file as necessary.

Management personnel have earned a Baccalaureate degree from an accredited college or university.

Analytical Chemists have earned a Baccalaureate Degree in Science or related fields from an accredited college or university.

Technical Staff have applicable training, including on the job training, and/or experience in related fields.

### 4.2 Analytical Methods

Analytical methods used for the analysis of environmental samples are described in a set of written instructions completely defining the procedure to be followed to process a sample and obtain an analytical result. An analytical method describes, as a minimum, the analytes for which it is valid, the matrix type, sample preparation, reagent and standards preparation, instrument calibration, and computations used to evaluate the analytical results. Standards and quality control sample requirements are also defined.

Analytical methods are either supplied by USAEC or, with approval, developed by DCL. The documentation for proposed methods development includes:

1. The submission of documentation to USAEC.
2. A statement of the problem.
3. A description of the technical approach to include specific details on procedures, solvents, instrumentation, etc.
4. An estimate of resources required (to include labor hours, funds and schedule).

When the testing of the analytical procedures has been successfully completed, the method is documented in the standardized USATHAMA method format. The format for documentation of all analytical methods is provided in Table 1. The format for data analysis is established by USAEC-provided statistical analysis computer software. Updates to the software are implemented upon receipt.

**Table 1.**  
**FORMAT FOR DOCUMENTATION OF METHOD CERTIFICATION**

- I. Summary**
  - A. Analytes
  - B. Matrix
  - C. General Method
- II. Application**
  - A. Tested Concentration Range
  - B. Sensitivity
  - C. Reporting Limit
  - D. Interferences
  - E. Analysis Rate
  - F. Safety Information
- III. Glassware and Chemicals**
  - A. Glassware/Hardware
  - B. Instrumentation
  - C. Analytes
  - D. Reagents and SARMS
- IV. Calibration**
  - A. Initial Calibration
  - B. Daily Calibration
- V. Certification Testing**
- VI. Sample Handling and Storage**
  - A. Sampling Procedure
  - B. Containers
  - C. Storage Conditions
  - D. Holding Time Limits
  - E. Solution Verification
- VII. Procedure**
  - A. Separations
  - B. Chemical Reactions
  - C. Instrumental Analysis
  - D. Confirmational Analysis
- VIII. Calculations**
- IX. Daily Quality Control**
  - A. Control Samples
  - B. Control Charts
- X. References**
- XI. Data**



The analytical method, once certified, is followed for all USATHAMA analyses. Instrumental conditions are optimized within the limits specified by method and documented by the analyst. Any deviation, other than the optimization of instrumental conditions, is pre-approved by USAEC before implementation.

All copies of USATHAMA-certified methods are individually numbered. Each distributed method copy must be signed for and dated. A comprehensive list of all distributed methods is kept by the Quality Assurance Coordinator.

#### 4.3 Method Certification

Before field samples may be analyzed by the laboratory, the methods of analysis must be certified. Certification for selected methods, accomplished under other USAEC contracts, may be determined by USAEC to be acceptable for the work performed under this contract for identical analytes and matrices. If analytes are required for a particular certified method in addition to those which have already been certified, the additional analytes are appended to the current certified method by following full certification procedures for the additional analytes. The current certified method standards, concentrations and analytical conditions are used to certify the additional compounds.

Some methods, including calibration of test and measurement equipment, do not require certification, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, USAEC will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, and project QC plans to describe exactly the procedures to be used. A copy of a proposed method must be submitted to the USAEC Chemistry Branch before it is used on any project.

The following methods do not require certification by the USAEC Chemistry Branch: temperature, conductivity, pH, oil and grease, hardness, asbestos, alkalinity (carbonate/bicarbonate/hydroxide), total organic carbon, biochemical oxygen demand, chemical oxygen demand, total dissolved solids, total suspended solids, totals solids, total petroleum hydrocarbons, salinity, and acidity.

##### 4.3.1 Written Method

A draft of the analytical method proposed for certification is submitted to USAEC for approval with the precertification performance data package.

##### 4.3.2 Standards

Standard Analytical Reference Materials (SARMs), provided by USAEC, are used in all method certification analyses. DCL obtains suitable, certified Reference Materials from the EPA or other commercial sources for analytes for which USAEC is not able to provide SARMs. Standard water and standard soil are used by DCL for all USAEC analyses done.

#### 4.3.3 Standard Water

Standard water samples are prepared by adding a known quantity of target analyte to a known volume of water. The volume of water is specific in the method being performed. All target analytes for the method are added. ASTM Type II grade water is used for all analyses. The method and reagents used to prepare spiking solutions are specified in the standardized methods.

#### 4.3.4 Standard Soil

Standard soil samples are prepared by adding a known quantity of target analyte to a known weight of selectively blended standard soil as provided by the Chemistry Branch of USAEC.

#### 4.3.5 Precertification Calibration

Before initiating method certification, precertification calibration is performed. DCL holds discussions with USAEC delineating anticipated environmental concentrations. The concentration range tested includes the Target Reporting Limit (TRL). Additional concentrations of calibration standards may be included for expanding the range of certification. Duplicate analyses are performed on all of the calibration standards.

The certified check standards are obtained from a source other than USAEC, whenever possible. In the absence of suitable commercially prepared mixtures, the DCL Quality Control Chemist prepares appropriate mixtures from certified pure stock reagents. The mixtures contain the analyte(s) of interest at concentrations near the high end of the certification range.

The calibration standard data is tabulated and graphed for analysis of Lack of Fit (LOF) and Zero Intercept (ZI), then submitted to USAEC for evaluation. The check standard results are required to fall within the acceptability limits defined by the originator.

#### 4.3.6 Certification

Certified methods meet the following conditions: The Target Reporting Limit (TRL) and the range of certification are selected in consultation with USAEC. A pre-certification analysis is performed and reported to USAEC, with a copy of the analytical method. Upon approval from USAEC, a Class 1, Class 1A, Class 1B, or Class 2 certification process is initiated. See Table 2.

Data derived from certification is processed using USAEC supplied software, and submitted to USAEC for evaluation. The method Certified Reporting Limit (CRL) and certified range are determined from this data evaluation.

Methods certified under previous editions of the USATHAMA Quality Assurance Program and determined by USAEC to be valid for current work do not require recertification.

All certification data are properly maintained in archive files.

#### 4.3.7 Method Modifications and Control

Any modifications, additions, or deletions proposed to any certified USATHAMA method must be submitted to USAEC for approval before such a change is made. Following approval, the revised method (with changes plainly noted) shall be distributed to appropriate laboratory personnel as described in DCL SOP-GLP-002, and the old method collected for retirement.

#### 4.4 Analyst Training

An analyst certifying a new method is qualified to perform that method during routine field sample analysis. An analyst who is required to perform on a procedure which has already been certified is required to satisfactorily analyze an appropriate set of quality control samples to demonstrate ability to perform the method. The demonstration sample data must pass current quality control criteria. Successful certification performance is reflected by an addition to the analyst's training file.

The analyst prepares all data records and a data package, as required for field sample analysis data. The data and the data package must be approved by Quality Assurance. The data and data package are maintained in archives.

**Table 2.**  
**NUMBERS AND CONCENTRATIONS OF CALIBRATION STANDARDS**  
**(LINEAR AND ZERO-INTERCEPT)**

**PRECERTIFICATION - CLASS 1**

Minimum Testing Range (MTR): 12 Standards + 1 Check Standard (SC)

Blank, \*0.5, 1, 2, 5, & \*10 TRL (Duplicate) + CS

MTR + 1 Order of Magnitude Extension: 18 Standards + 1 Check Standard (CS)

Blank, \*0.5, 1, 2, 5, 10, 20, 50, & \*100 TRL (Duplicate) + CS

MTR + 2 Orders of Magnitude Extension: 24 Standards + 1 Check Standard (CS)

Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, & \*1000 TRL (Duplicate) + CS

**PRECERTIFICATION - CLASS 1A**

Minimum Testing Range (MTR): 8 Standards

Blank, \*0.5, 2, & \*10 TRL (Duplicate)

MTR + 1 Order of Magnitude Extension: 12 Standards

Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate)

MTR + 2 Orders of Magnitude Extension: 16 Standards

Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate)

**PRECERTIFICATION - CLASS 1B**

Minimum Testing Range (MTR): 8 Standards + 1 Check Standard (CS)

Blank, \*0.5, 2, & \*10 TRL (Duplicate) + CS

MTR + 1 Order of Magnitude Extension: 12 Standards + 1 Check Standard (CS)

Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate) + CS

MTR + 2 Orders of Magnitude Extension: 16 Standards + 1 Check Standard (CS)

Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate) + CS

**PRECERTIFICATION - CLASS 2**

(Not Required)

**INITIAL CALIBRATION - CLASS 1**

Minimum Testing Range (MTR): 7 Standards + 1 Check Standard (CS)

Blank, \*0.5, 1, 2, 5, \*10, & \*10 TRL + CS

MTR + 1 Order of Magnitude Extension: 10 Standards + 1 Check Standard

Blank, \*0.5, 1, 2, 5, 10, 20, 50, \*100, & \*100 TRL + CS

MTR + 2 Orders of Magnitude Extension: 13 Standards + 1 Check Standard

Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, \*1000, & \*1000 TRL + CS

\* 10 percent to 25 percent Range Extension

**Table 2**  
**(Continued)**

**INITIAL CALIBRATION - CLASS 1A**

Minimum Testing Range (MTR): 5 Standards

Blank, \*0.5, 2, \*10, & \*10 TRL

MTR + 1 Order of Magnitude Extension: 7 Standards

Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL

MTR + 2 Orders of Magnitude Extension: 9 Standards

Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL

**INITIAL CALIBRATION - CLASS 1B**

Minimum Testing Range (MTR): 5 Standards + 1 Check Standard (CS)

Blank, \*0.5, 2, \*10, & \*10 TRL + CS

MTR + 1 Order of Magnitude Extension: 7 Standards + 1 Check Standard

Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL + CS

MTR + 2 Orders of Magnitude Extension: 9 Standards + 1 Check Standard

Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL + CS

**INITIAL CALIBRATION - CLASS 2**

Minimum Testing Range (MTR): 6 Standards

Blank and 1 TRL (Triplicate)

**DAILY CALIBRATION - CLASS 1/CLASS 1A/CLASS 1B**

Minimum Testing Range (MTR): 2 Standards

\*10 & \*10 TRL

MTR + 1 Order of Magnitude Extension: 2 Standards

\*100 & \*100 TRL

MTR + 2 Orders of Magnitude Extension: 2 Standards

\*1000 & \*1000 TRL

**DAILY CALIBRATION - CLASS 2**

Minimum Testing Range (MTR): 4 Standards

Blank and 1 TRL (Duplicate)

**Table 2  
(Continued)**

**CERTIFICATION - CLASS 1**

Minimum Testing Range (MTR): 9 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 12 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 15 Initial, 6 Daily

**CERTIFICATION - CLASS 1A**

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial

**CERTIFICATION - CLASS 1B**

Minimum Testing Range (MTR): 6 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 8 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 10 Initial, 6 Daily

**CERTIFICATION - CLASS 2**

Minimum Testing Range (MTR): 6 Initial

**INITIAL FIELD SAMPLE LOT - CLASS 1**

Minimum Testing Range (MTR): 9 Initial  
MTR + 1 Order of Magnitude Extension: 12 Initial  
MTR + 2 Orders of Magnitude Extension: 15 Initial

**INITIAL FIELD SAMPLE LOT - CLASS 1A**

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial

**Table 2**  
**(Continued)**

**INITIAL FIELD SAMPLE LOT - CLASS 1B**

Minimum Testing Range (MTR): 6 Initial  
MTR + 1 Order of Magnitude Extension: 8 Initial  
MTR + 2 Orders of Magnitude Extension: 10 Initial

**INITIAL FIELD SAMPLE LOT - CLASS 2**

Minimum Testing Range (MTR): 6 Initial

**ADDITIONAL FIELD SAMPLE LOT - CLASS 1/CLASS 1A/CLASS 1B**

Minimum Testing Range (MTR): 2 Daily  
MTR + 1 Order of Magnitude Extension: 2 Daily  
MTR + 2 Orders of Magnitude Extension: 2 Daily

**ADDITIONAL FIELD SAMPLE LOT - CLASS 2**

Minimum Testing Range (MTR): 4 Daily

## 5.0 SAMPLE HANDLING AND ANALYSIS

### 5.1 Sample Management

In most instances, DCL does not perform sample collection, but receives samples from designated field crews. Samples received by DCL are received by designated sample custodians. The protocols of sample management are delineated below.

#### 5.1.1 Sample Containers

As directed by USAEC, DCL will supply sample bottles and/or shipping coolers for use in the collection of field samples. A copy of DCL's "Field Sampling Information," to be used as guidance in sampling and in the completion of chains-of-custody, is included in the initial shipment of coolers to the field sampling site. All sample containers shall be cleaned before use according to the protocols specified in Appendix C. Use of commercially cleaned bottles is acceptable provided that cleaning is performed as specified in Appendix C or meets the requirements of the EPA's Contract Laboratory Program.

Generally, for water samples, this includes: septum-sealed glass vials for volatile compounds; amber glass bottles with Teflon-lined lids for organic constituents other than volatiles; and polyethylene bottles for inorganic analytes. Exceptions are noted in the certified method. For soil and sediment samples wide-mouth amber-glass bottles shall be used. Preservatives, as delineated in the DCL USATHAMA Analyte Summary (Appendix B), are provided (as necessary) with sample containers shipped to the field, for proper addition at the site.

#### 5.1.2 Sample Receipt

Samples are received at DCL by the designated Sample Receipt Officer (SRO), or his designee. At the time of receipt of a sample shipment, the sample shipping containers are opened and the samples are inspected. A Sample Receipt Form is initiated at this time. This form includes entries for date and time of receipt, airbill number, a record of the condition of seals on the shipping container and samples, documentation present, temperature and general condition of the shipment, and correlation of sample document and sample labeling information.

Any discrepancies between the samples and the documentation, including missing, broken, or damaged samples, will be reported to USAEC or its contractor within 24 hours.

The SRO or his designee signs the field chain-of-custody record at the time that the shipping container is opened. In the case of water samples, which do not usually require splitting, the SRO or his designee opens the shipping container and completes the sample inspection form and field chain-of-custody record. Sufficient copies of the field chain-of-custody record are made to allot one copy for each analytical procedure, plus one for moisture and one as a back-up.



#### 5.1.3 Sample Logging

The field chain-of-custody record is used by the Sample Receipt Coordinator (SRC) to initiate sample logging procedures. Initial logging entries include field sample number, date of receipt at DCL, analyses requested, and comments on sample condition at the time of receipt as noted on the Sample Receipt Record. These are recorded in both a computer based log and in a bound logbook. After sample lotting is completed, the USAEC sample identification number for each sample and analysis is entered into the logs.

#### 5.1.4 Sample Splitting

Following initial sample inspection, the SRC splits the samples into the required number of aliquots (one for each analytical procedure, one for moisture if the sample is a soil, and a large portion for back-up). The SRC properly labels the aliquots with the field sample identification number and the method of analysis, and relinquishes custody of the sample aliquots to the SRC.

#### 5.1.5 Sample Lotting and Labeling

The number of samples which can be analyzed by a given method on a single day, as determined by the rate-limiting step in the analytical scheme, is designated as a "lot". The samples in a lot are labeled with a USAEC sample identification number consisting of a three letter lot code and individual three number sample designations (e.g. AAA001, AAA002). As split sample aliquots for a particular analytical procedure are received by the SRC, they are given the next alphabetical lot designation in sequence. Samples received and split at various times are grouped together in the same lot such that sample holding times are not jeopardized. The unique sample number is written in black permanent marker on white laboratory labeling tape, which is prominently placed on each sample container.

Quality control (QC) samples are a part of every lot, and are spiked according to the specific method requirements. The QC samples are provided upon request of the analyst.

#### 5.1.6 Sample Storage

Samples are stored in a location appropriate to the holding requirements of the requested analytes. Heat-sensitive, light-sensitive, radioactive, or other samples having unusual physical characteristics or requiring special handling, are properly stored and maintained.

### 5.2 Chain-of-Custody

DCL maintains chain-of-custody records for all USAEC samples received at the laboratory.

A copy of applicable field chain-of-custody records is maintained with each sample lot. In addition, each lot of samples is maintained under a separate laboratory chain-of-custody record. The chain-of-custody includes unique sample number(s), date and time, source of sample(s), analyses required, signatures of relinquishing and receiving entities, and any other pertinent information. Copies of DCL's field and in-house chains-of-custodies for USAEC projects are provided in Appendix D.

### 5.3 Sample Handling Procedures

After samples have been received, split, and lotted, those not requiring extraction procedures are transferred to a central walk-in cold storage area. They are stored in this area until they are scheduled for analysis. Samples not requiring extraction procedures are prepared for analysis, within the required holding times, by the analyst or by a technician working under the direction of the analyst. These samples are usually analyzed within hours after preparation.

Samples which require extraction, distillation, or digestion procedures are prepared for analysis by the appropriate Inorganic or Organic Sample Preparation groups after lotting procedures have been completed. Extracts or distillates are stored in refrigerators in appropriate analytical areas of the laboratory.

The samples and extracts are maintained in their designated lots and under chain-of-custody, at all times. Separate preparation logbooks are maintained by the sample preparation groups to document sample handling.

### 5.4 Toxicity Characteristic Leaching Procedure

Samples which require Toxicity Characteristic Leaching Procedure (TCLP) are split and assigned a unique three-letter lot code. Chains-of-custody for these samples are signed off in the same manner as other samples requiring a certified USATHAMA analysis. At the same time, chains-of-custody are printed (but not "initiated") for all prospective analyses to be generated from the TCLP leachate(s).

Once the original sample has been satisfactorily leached, both the chain-of-custody and any remaining original sample are transferred to Long Term Storage. The chains-of-custody for all generated leachates are now initiated by TCLP personnel. These leachates (along with their chains-of-custody) are stored and handled as any other USAEC samples which have been prepared for analysis.

The chains-of-custody for the original sample and the leachates are cross-referenced to facilitate traceability.

### 5.5 Holding Times

The holding times specified in DCL's USATHAMA Analyte Summary (Appendix B) are adhered to for all USAEC samples, extracts, distillates, and digestates.

## 5.6 Sample Analysis

### 5.6.1 Standards

Analytical standards are prepared either from Standard Analytical Reference Materials (SARMs) or Interim Reference Materials (IRM) supplied by USAEC, or from standard materials obtained by DCL from the EPA, the National Institute of Standards and Technology (NIST), or other commercial sources. Secondary standard materials may be used when SARM materials are available in only limited quantity. The secondary standards, which must be positively identified with an estimation of purity, are referenced to SARMs and periodically checked against them.

Standard materials procured from commercial sources other than USAEC, the Environmental Protection Agency (EPA), or the NIST are considered as "off-the-shelf" materials. The purity and identity of these materials is established from both analysis documentation supplied by the vendor and DCL analytical data. Materials are characterized by two independent methods whenever possible, including, but not limited to IR, GC, GC/MS, HPLC, and other inorganic techniques.

Metals are traceable to NIST, whenever possible. "Off-the-shelf" materials are characterized against EPA or NBS known standards whenever possible. All SARMs are stored in the quality control laboratory, under controlled access conditions. Generally, organic compounds are stored under refrigeration, while metals solutions are stored at room temperature.

### 5.6.2 Solutions

Analytical standard working solutions are normally prepared by the analyst performing the analysis, in accordance with the protocol defined in the approved analytical method. In some analytical procedures, a designated analyst prepares the standards, while other analysts carry out the procedure.

As new or replacement standard solutions are prepared, they are validated against either the previously used standard, a commercially prepared quantitative standard, or a standard prepared by another analyst for the purpose of validation.

Although validation acceptance criteria are established for each analytical method, protocol guidelines for acceptance of a new solution is that it is found, by analysis, to be within  $\pm 5\%$  of the target value. All validations are documented either in the analyst's notebook or in a standards preparation logbook unique to USAEC and the analytical area using the standards.

### 5.6.3 Sample Preparation

Soil and water field samples are prepared for analysis according to the protocol defined in the analytical method for the specific analyte(s) being analyzed. Procedures for the preparation of mixed-matrix field samples, such as sediment, sludge, sewer, or lake-bottom samples, are discussed with USAEC on a case-by-case basis.

#### 5.6.4 Instrument Calibration

The USATHAMA QA Program delineates, in detail, the requirements for instrument calibration for precertification, full method certification, initial calibration for analysis work, and daily calibration during sample analysis. DCL has implemented these guidelines for all USAEC work, as follows. Also see Section 4.3.6 (Certification) for additional details.

Instruments are tuned, as applicable, and the required number and concentrations of standards are analyzed daily with each lot of samples. Calibration criteria are either passed or corrective action is pursued by the analyst. If daily calibration criteria are not met, then initial calibration procedures are instituted to bring the analytical system back into calibration.

#### 5.6.5 Initial Calibration

During initial calibration, a minimum of one blank and five calibration standards (Class 1) or one blank and three calibration standards (Class 1A and Class 1B) that bracket the certification testing range is analyzed singularly on one day. The concentrations of the calibration standards, in the solvent that results from all the preparation steps of the method, take into account any concentration steps that are part of the method. Concentrations in the solvent correspond to those in an environmental matrix as if the method preparation steps had been performed.

In addition to the initial calibration standards, Class 1 and 1B methods require the analysis of calibration check standards (Section 5.6.7). During a Class 1 or Class 1B initial calibration, a calibration check standard is analyzed at the completion of calibration. If the method requires what could be an initial calibration each day analysis is performed, then the calibration check standards are analyzed once a week rather than each day.

If the results of the calibration check standard are not acceptable, immediate reanalysis of the calibration check standard is required. If the results of the reanalysis still exceed the limits of acceptability, the system is considered to have failed calibration. Sample analysis is halted and will not resume until successful completion of initial calibration. Corrective actions taken to restore initial calibration are documented in the analysts' notebook.

#### 5.6.6 Daily Calibration

Calibration standards are analyzed each day to verify that instrument response has not changed from previous calibration. Each day before sample analysis, the highest concentration standard is analyzed. The response must fall within the required percentage or two standard deviations of the mean response for the same concentration, as determined from precertification, certification, and prior initial/daily calibrations. If the response fails this test, the daily standard is reanalyzed. If the response from the second analysis fails this range, initial calibration is performed before analyzing samples.

Each day after sample analyses are completed, the highest concentration standard is analyzed. If the response is not within the required percentage or two standard deviations of the mean response from precertification, certification, and prior initial/daily calibrations, the daily standard shall be reanalyzed. If the response from the second analysis fails the range, the system is considered to have failed calibration. Initial calibration is performed and all samples analyzed since the last acceptable calibration are reanalyzed.

For non-linear or non-zero-intercept calibration curves, daily calibration consists of analysis of the low, middle, and high standards at the beginning of the day. When sample analyses are completed at the end of the day, the low and high standards are analyzed. Instrument responses for each concentration determination must fall within two standard deviations of the mean response, as described previously, for the appropriate standard. For calibrations fitted by the quadratic equation, a minimum of four standards over the certified range are required and the highest level standard analyzed at the end of the day. For all other equations, one more standard than needed to meet the degrees of freedom for any lack-of-fit is required, as a minimum.

#### 5.6.7 Calibration Check Standards

Calibration check standards are required for all Class 1 and 1B methods and are analyzed during precertification and with each initial certification. The calibration check standard contains all analytes of interest for the method in question at a concentration near the upper end of the calibration range. Results of the calibration check standards shall fall within the limits of acceptability as described below:

##### CASE 1.

A certified check standard is available from the EPA or some other source with both the true value and limits of acceptability specified by the supplier. The results must fall within the limits specified by the supplier, or  $\pm 10$  percent for inorganics,  $\pm 25$  percent for organics, whichever is less.

##### CASE 2.

A certified check standard is available from the EPA or some other source with a true value specified but without limits of acceptability. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

##### CASE 3.

If no certified check standard is available, the contractor laboratory shall prepare a check standard using a second source of reference material. This standard shall be prepared by a different analyst than the one who prepared the calibration standard. If weighing of the material is required, a different balance should be used, if possible. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

##### CASE 4.

If there is only one source of reference material available, then the calibration and calibration check standards must be prepared from the same material. The standards shall be prepared by different analysts. If weighing is required, different balances should be used, if possible. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

For all cases listed above, after the seventh acceptable calibration check standard, the limits of acceptability are +/- two standard deviations, as determined from the first seven points.

For multi-analyte methods, the calibration check standard contains all analytes of interest. For the check standard to be deemed acceptable at least 2/3 of the analytes must meet the limits of acceptability as defined above (also see Table 3). In addition, if a single analyte falls outside the limits of acceptability for two consecutive times, then the calibration check standard is deemed unacceptable. If a calibration check standard is not acceptable, the procedures detailed above are followed.

**Table 3.**  
**MINIMUM NUMBER OF IN-CONTROL POINTS**  
**FOR MULTI-ANALYTE METHODS**

<u>Required Control</u> <u>Analytes Per Method</u>	<u>Required Number of</u> <u>Data Values Falling</u> <u>Between the UCL and LCL</u>
1	1
2	2
3	2
4	3
5	4
6	4
7	5
8	6
9	6
10	7
11	8
12	8
13	9
14	10
15	10
16	11
17	12
18	12
19	13
20	14
21	14
22	15
23	16
24	16
25	17

#### **5.6.8 Analytical Procedures**

All field samples are analyzed according to approved, laboratory certified USATHAMA analytical methods. All deviations shall be approved by USAEC prior to implementation. These deviations are also documented in the analyst's notebook.

#### 5.6.9 Second-Column Confirmation

In several GC and HPLC methods (e.g., organochlorine pesticides and explosives), the presence of compounds is routinely confirmed on a second column. The confirmation is usually performed on the basis of a Class 2 certification. Confirmation does not necessarily have to be performed within holding times, but must be accomplished within ten (10) days of sample analysis.

### 5.7 Data Handling

Although the primary emphasis of the USATHAMA QA Program is the control of sample analysis and the handling of data, record keeping maintains its importance in the overall assessment of the production of quality of data and is used in part to document the control of sample analysis. The degree of rigor used in documenting sampling and analysis activities cannot be understated. All activities require extensive documentation and special handling protocols. All activities are to be performed under chain-of-custody procedures. Particularly in these situations, the attitude is: "If you didn't write it down, you didn't do it."

For most USAEC projects, this degree of documentation is required. For some projects, documentation in the form of an EPA CLP package is required. In any case, the records described in this Quality Assurance document shall be maintained and will be available for inspection by USAEC.

#### 5.7.1 Data Reduction

Generally, data have been collected during the analysis of samples either into computer based data files or onto hard copy sheets, which, in turn, are either machine generated or hand written. All of the data are eventually compiled in computer files. The data pertaining to analytical standards are either compared to the most recent initial calibration curve, in the case of a daily calibration, or used to generate new initial calibration curves, in accordance with those generated during pre-certification. The appropriate standard curve is used to evaluate the field sample data to determine the amount of analyte present. Finally, all of the computer generated calculations are generated as hard copy output.

#### 5.7.2 Data Validation

Initial data validation is accomplished during data collection through the use of quality control samples and calibration check standards. Errors detected through a review of these monitors by Quality Assurance during analysis are corrected during the data collection phase of the analysis. Only analytically valid data are processed further.

Following an analyst's computer-based reduction of data and production of a numerical results report, the entire assemblage of data is given to a peer analyst for review and validation. The peer analyst checks that the analytical method was followed, that there are no errors in the transcription of data, that the best-fit curve was used, and that the numerical report of data contains no calculation or transcription errors.

The data package is then reviewed by the appropriate Group Leader or Section Manager. The data report is particularly scrutinized to assure that all reported data values are in the proper range or have dilution factors, that the method has been carefully followed, that instrumentation was properly tuned or calibrated, and that the instrumental data was properly interpreted. A general review of the data package is also made to assure that all required documentation is present.

The final step in data validation is the review by Quality Assurance. The content of each data package is closely checked for errors or omissions that would negatively impact on the admissibility of the data in litigation proceedings. Corrective action is initiated and documented as outlined in section 10.0.

### 5.7.3 Data Reporting

The results for samples analyzed for USAEC projects are entered into the USAEC-provided software program (IRDMS). Data created using the IRDMS can then be electronically transmitted to USAEC Via Potomac Research Inc. (PRI), or a diskette together with hard copy printouts can be submitted.

Data is entered on a coding form by the analyst, which is verified by the peer checker and, group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USAEC software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to USAEC. DCL retains a copy diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data is also reviewed by Data Entry for completeness before release.

All data packages are archived at DCL until a task or delivery order at a particular installation is complete. At that time, all pertinent documentation filed in appropriately-labeled boxes is delivered either to USAEC directly, or to the prime contractor responsible for final review of the data packages. In the second case, the prime contractor is responsible for the delivery of DCL data boxes to USAEC.



## 6.0 ANALYTICAL SYSTEM CONTROLS

### 6.1 Sample Control

As discussed in the section of this QA Plan on Sample Management, DCL is not generally responsible for the collection of samples from sites in the field. However, DCL efforts in sample control may extend into field sample collection. As directed by USAEC or the prime contractor, DCL provides proper sample collection bottles, sample preservatives, labeling material, sample shipping containers (coolers), and technical assistance to field sample collection crews. DCL also works in concert with USAEC or the prime contractor on sample shipping and receiving.

Samples received at DCL are under the control of Sample Receipt personnel from receipt at the lab to acceptance by an analyst for extraction or preparation. Samples are not released for processing until all documentation is completed and the samples are properly lotted and labeled. Holding times are closely monitored by the analysts, Sample Receipt and laboratory management.

DCL Project Managers communicate regularly with USAEC and/or other involved prime contractors to alleviate sample shipping, holding time, and analysis difficulties.

### 6.2 Document Control

Document control is primarily the responsibility of Quality Assurance. Sample documents generated in the field during sample collection and shipping are maintained in QA files. Laboratory chain-of-custody records, sample receipt and tracking records, data reporting forms and analysis data packages, and corrective action records are maintained by Quality Assurance. On a schedule determined by contract requirements, QA also archives or otherwise controls all bound notebooks and logbooks containing data pertinent to USAEC work.

### 6.3 Quality Control Samples

Quality control chemists within the Quality Assurance Section of DCL prepare most of the quality control samples required during sample analysis. These samples are prepared from USAEC-supplied SARM and IRM stocks, and other reference materials. Other reference materials include EPA, and NIST standard materials, and "off-the-shelf" materials. "Off-the-shelf" materials are analyzed by DCL, with positive identification and estimate of purity, with EPA standard reference materials, where possible, using at least two different methods.

Quality control stock and dilute working solutions are prepared and maintained separately from those used by analysts as standards. Exceptions to this procedure are made only when primary stock material is in very short supply, or when the primary solution is unstable. In these cases, the same primary solution is used to prepare separate dilute working solutions. Samples are prepared in accordance with parameters defined in each analytical method. These parameters include the control analytes, the concentration levels at which the analytes should be spiked, control sample matrix, spike equilibration time, and procedures for preparation of the sample for analysis.

Quality control samples which are not regularly prepared by the quality control chemists include surrogate spiking solutions and spiked samples required in the GC/MS methods for volatile and semi-volatile organic compounds. These surrogate preparations are handled by the GC/MS Group and the Extraction Group, respectively.

Quality control samples are included in every lot of USAEC samples, as required in the USATHAMA QA Program and specified in each certified analytical method. The control samples are processed through the entire analytical method and quantitated on the same calibration curve as the field samples. The results for the quality control samples are evaluated first by the analyst, and then by Quality Assurance, to determine their acceptability.

Calibration check standards are prepared by someone other than the person preparing the standards. Calibration check standards are analyzed at the time of an initial calibration, or once per week when routine initial calibrations replace daily calibrations. The analysis results must meet the criteria established by their originator.

#### 6.4 Control Charts

For Class 1, Class 1A, and Class 1B certified methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts is not adjusted for accuracy. Control charts are not used with Class 2 certified methods.

Control charts include the analyte, method number, DCL laboratory code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USAEC-supplied computer control chart program:

1. Single-Day X-Bar Control Chart (High Spike Conc.)
2. Single-Day Range Control Chart (High Spike Conc.)
3. Three-Day X-Bar Control Chart (Low Spike Conc.)
4. Three-Day Range Control Chart (Low Spike Conc.)

In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the x-axis;
- Percent recovery (for X-bar control charts), or range (for R control charts) along the y-axis;
- Upper control limit (UCL);
- Upper warning limit (UWL);
- Mean;
- Lower warning limit (LWL), on X-bar charts; and
- Lower control limit (LCL), on X-bar charts.

For some analytes specified by USAEC, warning limits on X-bar charts are deleted and replaced by modified control limits based upon data quality specifications.

#### 6.4.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of certification data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are accepted, based upon the recoveries established during certification and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98% confidence level. If the datum is not classified as an outlier by the test, the point is included in updating the control chart limits. If the datum is classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. Charts are then updated with newly calculated control limits and all points plotted.

#### 6.4.2 Three-Point Moving Average

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above (Section 6.4.1) for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

#### 6.5 Out-of-Control Conditions

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each day of analysis. Processing of additional lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: A value outside the control limits or classified as outlier by statistical test; A series of seven successive points on the same side of the mean; A series of five successive points going in the same direction; A cyclical pattern of control values, or; Two consecutive points between the UWL and UCL or the LWL and LCL

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to 2/3 of the control analytes meet control criteria. Of the remaining control analytes (less than 1/3 possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than 2/3 of the control analytes are classified as in control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also section 10.0.) Field sample data effected by the situation are evaluated and reanalyzed as necessary.

When a method is determined to be out of control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

## **7.0 PREVENTATIVE MAINTENANCE**

All analytical instrumentation used at DCL is maintained to provide consistent, high-quality performance. Most instruments are maintained by the manufacturer, under contract. Instrument service records and maintenance calibrations are maintained by the appropriate section and in a logbook unique for each instrument.

The primary objective of the instrument maintenance program is to assure the quality of the analytical data generated by the instrument. While there are analytical systems which require absolute calibration, such as balances, the majority of analytical systems used by DCL for the analysis of USAEC samples are calibrated at the time of use by the analyst. This is accomplished through generation of a chemical calibration curve, based upon instrument response verses analyte concentration. This curve is used to evaluate field sample data through instrument responses.

Major instrument systems which are calibrated on an "as used" basis are maintained under either an "on call" or a preventative maintenance contract with the manufacturer. Preventative maintenance is scheduled in each instrument contract. When an instrument cannot perform to specifications and DCL technicians cannot return it to specification, a contracted repair service (usually the manufacturer) is called.

Instrument systems which must maintain an absolute calibration, such as analytical balances, are serviced under contract with the manufacturer, usually on an annual basis. Balances are also checked, on at least a weekly basis, for accuracy by Quality Assurance, using NIST-traceable weights. Temperatures of freezers, refrigerators, and walk-in coolers are recorded every working day by QA. When temperatures are noted outside the acceptable range, appropriate personnel are notified for correction. Ovens are calibrated and their temperatures maintained regularly by the appropriate section personnel.

## 8.0 RECORDKEEPING

### 8.1 Laboratory Notebooks

Bound, sequentially-numbered laboratory notebooks with pre-numbered pages are utilized by all analysts for analytical recordkeeping. Notebooks are generally issued to and used by an individual analyst. Any loose sheets of data which must be included in a notebook are securely taped into the notebook and signed and dated across the edges, halfway on the inserted sheet and halfway on the notebook page. Each data page is signed and dated by the analyst entering data on that page, as well as reviewed, signed, and dated by a witness. All entries are required to be in black ink. Corrections are made by a single strikeout, which is dated and initialed.

### 8.2 Logbooks

#### 8.2.1 General

Individual logbook entries are signed and dated by the analyst or technician making the entry. These notebooks include, for example, instrument use and maintenance/calibration logs, pH logs, sample moisture determination logs, and sample receipt logs.

Recordkeeping for sample receipt is discussed under the Sample Management Section 5.1.

#### 8.2.2 Standards

A bound logbook is maintained for all analytical reference materials used for USAEC work. The record includes the date of receipt, preparer, source, purity, composition, storage requirements, and expiration date, if applicable. Characterization data for purchased reference material is also included.

The preparation of working standards from reference materials is recorded in a bound logbook. This logbook may be of general use by several analysts for USAEC standards preparation, or an individual analyst's notebook, as for preparation of standards used for a single analytical run associated with a single lot of samples.

#### 8.2.3 Instrument

Instrument maintenance records and, where applicable, instrument tuning and calibration data, are maintained in instrument specific logbooks. Actual analytical conditions pertaining to an individual lot analysis are recorded in the analyst's notebook, along with other pertinent analytical information.

### 8.3 Hard-Copy Output

Hard-copy output, (e.g., chromatograms and computer generated data evaluations) is labeled with date, time (where applicable), analytical method, sample numbers, the name or initials of the analyst generating the output, and other pertinent information. Storage of hard-copy output is with related analytical data pertaining to an individual lot analysis. All such data, comprising a complete record of an analysis, are compiled into one or more envelopes for archiving. The envelopes are properly labeled with the lot designation, method of analysis, matrix, analyst, analyst's notebook, and date of completion. When samples from multiple sites or projects are grouped together in a single lot, the data pertaining to each site are compiled (or copied) and stored separately, as directed by USAEC. All copies indicate the location of the original data.

### 8.4 Data Package Preparation

In general, all data should be maintained in two separate locations, the data package and the laboratory notebook(s).

Records to be contained in the data package should include, but are not limited to the following:

- Optimized instrumental conditions
- Original chromatograms, strip charts, and/or other instrument output
- Original chain-of-custody form and carrier transmittal documents
- All hardcopy GC/MS outputs
- Expanded scale blow-up of manually integrated peak(s).
- All data sheets or other pre-printed forms used by the contractor or laboratory.
- Copies of all relevant notebook pages. This should include preparation of standards, calibration, sample preparation/extraction, moisture determinations, calculations, and any other relevant comments.

Each data package should contain all information related to one lot for one installation. In cases where a lot has samples from more than one installation, then the information should be copied and placed in separate packages for each installation. In those packages which receive copies, the location of the original material should be identified.

Each data package should contain a contents and approval checklist. This should identify all materials which must be placed into the data package. This list should also list reviewer's names, dates of review, provide space for comments, notes, and corrective actions.



It is the responsibility of the contractor laboratory to review data packages for both content and correctness.

Included in the data package should be a discussion on the observations on the data contained in that data package. This discussion shall include, but not be limited to, observed matrix effects, blank results, control problems, deviations from approved SOPs, digressions from normal practices (i.e., manual integrations) and reasons thereof, etc. The impact on the usability of the data shall be discussed. Explanations on the use of the applicable flagging codes shall be provided.

A detailed SOP is currently in development at DCL.

## **9.0 AUDITS**

DCL facilities are always available for any required audits, announced or unannounced, by USAEC representatives.

The DCL Quality Assurance Coordinator conducts internal audits of critical functions within the laboratory, including verification that record keeping procedures are adequate, verification that general good laboratory practices, analytical methods and standard operating procedures are being followed, and continual assessment of quality control sample results. A summary of such audits is available for review at the laboratory. Internal audits shall be conducted by DCL QA personnel at a minimum rate of twice per month.

## 10.0 CORRECTIVE ACTION

When, as a result of audit procedures or the analysis of quality control samples, the analytical or other laboratory systems are found to be unsatisfactory, a corrective action is initiated. The unsatisfactory situation may be either immediate or long term in nature. Immediate short term problems may include unsatisfactory performance on quality control samples (which may be more involved than simply out-of-control data), errors or omissions in the compilation of the data package, or other problems peculiar to a single lot of samples. Long-term problems include trends or cycles in quality control sample analysis data, standard and solution preparation control, staff training in analytical and quality control procedures, or other problems which affect several analytical methods or multiple lots of samples.

To enhance the timeliness of corrective action and thereby reduce the generation of unacceptable data, problems identified by assessment procedures are resolved at the lowest possible management level. Problems that cannot be resolved at this level are reported to the Quality Assurance Coordinator (QAC) for resolution. The QAC determines the management level at which the problem can best be resolved, and notifies the appropriate manager. Weekly progress reports detail all problems and subsequent resolutions.

Steps included in the corrective action system include:

1. Defining the problem;
2. Assigning responsibility for problem investigation;
3. Investigating and determining the cause of the problem;
4. Assigning responsibility for problem resolution; and
5. Verifying that the resolution has corrected the problem.

Problems requiring corrective action may not be easy to identify or define. The situation may not be producing out-of-control data, but simply producing data not of the quality desired. The project manager, section managers, analysts, and the quality assurance staff combine efforts in solving long-term unsatisfactory situations.

All corrective actions are documented by Quality Assurance. Final corrective action reports, which relate to a particular lot analysis, are included in the data package for that lot.

## **11.0 QUALITY CONTROL REPORTS**

DCL provides weekly quality assurance evaluation reports to USAEC, in conjunction with weekly interim technical reports from project management. The QA reports include charts and tables of quality control data, a control chart checklist delineating contracts and lots, and copies of Corrective Action Reports (CARs). These CARs include explanations of analytical or quality control problems and discussions of the corrective actions taken to alleviate those problems. Observations of data trends or situations which could develop into problems are also discussed in this report, as well as preliminary acceptance or rejection of analytical data.